

**United States Court of Appeals**  
*for the*  
**Federal Circuit**

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CORNING INCORPORATED,

*Appellant,*

— v. —

DSM IP ASSETS B.V.,

*Appellee.*

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APPEAL FROM THE UNITED STATES PATENT AND TRADEMARK OFFICE  
PATENT TRIAL AND APPEAL BOARD NO. IPR 2013-00045

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**BRIEF FOR APPELLANT**

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October 21, 2014

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## **CERTIFICATE OF INTEREST**

Pursuant to Federal Circuit Rule 47.4, counsel for Appellant Corning

Incorporated certifies the following:

1. The full name of every party represented by the undersigned counsel in this case is: Corning Incorporated.
2. The name of the real party in interest represented by the undersigned counsel in this case is: Corning Incorporated.
3. All parent corporations and any publicly held companies that own 10 percent or more of the stock of the party represented by the undersigned counsel are:  
  
None.
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**TABLE OF ABBREVIATIONS**

<b>Parties</b>	
Corning	Petitioner-Appellant Corning Incorporated
DSM	Patent Owner-Appellee DSM IP Assets B.V.

<b>Terms</b>	
'189 Patent	U.S. Patent No. 6,298,189 to Szum et al.
'666 Patent or '666 Pat.	U.S. Patent No. 6,339,666 to Szum et al. (Ex. 1001)
A__	Joint Appendix at page(s), with (column:lines) for patents
Appeal	Federal Circuit Case 2014-1730 captioned <i>Corning Incorporated v. DSM IP Assets B.V.</i>
Coady	U.S. Patent No. 5,219,896 to Coady et al. (Ex. 1004)
Corning Reply	Corning's Reply to Patent Owner Response filed November 27, 2013 in IPR2013-00045 (Paper 61)
Court	United States Court of Appeals for the Federal Circuit
Decision Instituting IPR	Decision Institution of <i>Inter Partes</i> Review 37 C.F.R. § 42.108 by the PTAB on May 13, 2013 in IPR2013-00045 (Paper 11)
Final Decision	Final Written Decision 35 U.S.C. § 318(a) and 37 C.F.R. § 42.73 issued by the PTAB on May 9, 2014 in IPR2013-00045 (Paper 92)
IPR	<i>Inter Partes</i> Review
Jackson	U.S. Patent No. 4,900,126 to Jackson et al. (Ex. 1005)
Motion to Amend	Patent Owner's Motion To Amend Under 37 C.F.R. § 42.121 filed August 27, 2013 in IPR2013-00045 (Paper 44)
Petition	Corning's Petition for <i>Inter Partes</i> Review of the '666 Patent filed November 15, 2012 in IPR2013-00045 (Paper 2)
PO Response	Patent Owner's Response Under 37 C.F.R. § 42.120 to Corning's Petition for <i>Inter Partes</i> Review of Claims 1–20 of the '666 Patent filed August 27, 2013 in IPR2013-00045 (Paper 43)

<b>Terms</b>	
Preliminary Response	Patent Owner's Preliminary Response under 37 C.F.R. § 42.107 to Petition for <i>Inter Partes</i> Review of the '666 Patent filed February 21, 2013 in IPR2013-00045 (Paper 10)
PTAB	The Patent Trial and Appeal Board of the United States Patent and Trademark Office
Shustack	U.S. Patent No. 5,352,712 to Shustack (Ex. 1003)
Sufficient Adhesion Limitation	Limitation in all claims of the '666 Patent, wherein it recites "sufficient adhesion to said glass fiber to prevent delamination in the presence of moisture and during handling"
Szum	WO 95/15928 to Szum (Ex. 1002)
PTO	United States Patent and Trademark Office

All emphasis in this brief is added unless otherwise indicated.

## STATEMENT OF RELATED CASES

Pursuant to Federal Circuit Rule 47.5, counsel for Corning states that there is no other appeal in or from the same civil action or proceeding in the lower court or body that was or is before this or any other appellate court.

The following cases currently pending in this Court may directly affect, or be directly affected by, the Court's decision in this Appeal, which pertains to the '666 Patent. The '189 Patent, also owned by DSM, is related to the '666 Patent; the application from which the '666 Patent issued is a continuation of the application from which the '189 Patent issued. The '189 Patent is the subject of both IPR2013-00048 and IPR2013-00049, both of which are entitled *Corning Incorporated v. DSM IP Assets B.V.* The Final Decision for the '666 Patent was issued on the same day as the final decisions in IPR2013-00048 and IPR2013-00049. On September 8, 2014, Corning timely filed notices of appeal with this Court of the final decisions in IPR2013-00048 and IPR2013-00049.

The appeals for IPR2013-00048 and IPR2013-00049 involve similar or overlapping issues as this Appeal. For this reason, Corning respectfully requests that this Appeal be treated as a companion case to the appeals in IPR2013-00048 and IPR2013-00049. As of the filing of this brief, no docket numbers had been assigned by this Court for the appeals in IPR2013-00048 and IPR2013-00049.

On August 21, 2014, Corning commenced a declaratory judgment action against DSM and DSM Desotech, Inc. in the United States District Court for the District of Delaware, Case No. 14-cv-01081, entitled *Corning Incorporated v. DSM Desotech, Inc. and DSM IP Assets B.V., inter alia*, seeking a declaration that Corning does not infringe the '666 Patent and the '189 Patent, and that the '666 Patent and the '189 Patent are invalid. In addition, on or about October 16, 2014, DSM and DSM Desotech, Inc. commenced an action against Corning in the United States District Court for the Northern District of Illinois, Case No. 14-08111, entitled *DSM Desotech, Inc. and DSM I.P. Assets B.V. v. Corning Incorporated*, alleging, *inter alia*, that Corning will, has been and/or is directly infringing and/or inducing infringement of and/or contributorily infringing claims of the '666 Patent and the '189 Patent.

Other than the aforementioned cases, there are no other cases known to counsel to be pending in this or any other court that will directly affect or be directly affected by this Court's decision in the Appeal.

**STATEMENT REQUESTING ORAL ARGUMENT**

Oral argument is respectfully requested.

## STATEMENT OF JURISDICTION

This Appeal arises from the following action in the PTAB: *Corning Incorporated v. DSM IP Assets B.V.*, IPR2013-00045. This action relates to claims 1–20 of the '666 Patent.

The PTAB entered a Final Decision on May 9, 2014, rejecting Corning's challenge to claims 1–20. This Appeal involves the Final Decision and all findings, orders, decisions, rulings, and opinions underlying the Final Decision.

The PTAB had subject matter jurisdiction over the underlying case pursuant to 35 U.S.C. §§ 6(b)(4) and 316(c). This Court has jurisdiction pursuant to 35 U.S.C. § 141(c) and 28 U.S.C. § 1295(a)(4)(A). Corning timely filed a Notice of Appeal on July 8, 2014. No cross appeal was filed by DSM.

### **STATEMENT OF THE ISSUES**

- (1) Did the PTAB err in arriving at an unduly narrow claim interpretation, rather than the broadest reasonable interpretation of the Sufficient Adhesion Limitation and, thus, err in concluding Corning did not establish by a preponderance of the evidence that claims 1–20 of the '666 Patent should be cancelled as unpatentable?
- (2) Did the PTAB err in its refusal to accept DSM's non-contingent request to cancel claims 1 and 10 of the '666 Patent?

## STATEMENT OF THE CASE

### A. Introduction

Independent claims 1 and 10 of the '666 Patent recite a “composition for coating an optical fiber,” which after radiation cure has a combination of four recited properties: (a) a specific fiber pull-out friction value range; (b) a specific crack propagation value range; (c) a specific glass transition temperature range; and (d) “sufficient adhesion to said glass fiber to prevent delamination in the presence of moisture and during handling.” A70 at claims 1 and 10. This last property, which is referred to as the “Sufficient Adhesion Limitation,” is the subject of this Appeal. In particular, the Court must determine the correct broadest reasonable interpretation for the Sufficient Adhesion Limitation.

The specification of the '666 Patent describes tests for determining numerical value ranges for all of the properties recited in independent claims 1 and 10 *except* “sufficient adhesion.” The '666 Patent describes three different tests that relate to the adhesion between a radiation-cured coating and glass: a water soak delamination test (A50 at 27:32-47), a “dry” adhesion test at 50% relative humidity (A50-51 at 28:57-29:25), and a “wet” adhesion test at 95% relative humidity (A50-51 at 28:57-29:25). However, neither the Sufficient Adhesion Limitation language in the claims, nor the text of the specification, dictates or suggests whether one or



more of these tests should be performed to assess whether a particular coating composition meets the limitation.

DSM, in prosecuting claims 1 and 10, could have avoided this indefiniteness problem by drafting the claims to explicitly identify a test to be performed for the fourth property.<sup>1</sup> However, DSM chose not to do so and, instead, left the public to guess what the Sufficient Adhesion Limitation means. Likewise, in the specification, DSM could have explicitly defined both the meaning of the Sufficient Adhesion Limitation (*i.e.*, objective criteria to assess what falls within the claim scope and what does not) and which of the three adhesion-related tests should be performed. However, again, DSM provided the public with no clear direction.

Without any guidance or limitation on the proper test to determine the Sufficient Adhesion Limitation, Corning's choice of the wet adhesion test from the specification was both reasonable and appropriate. Moreover, the wet adhesion test is the only one of the three tests listed that (1) measures "adhesion" in the presence of a significant amount of "moisture" (hence the name "*wet*" adhesion test) and (2) involves handling, both of which are required by the Sufficient Adhesion Limitation.

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<sup>1</sup> While "indefiniteness" under 35 U.S.C. § 112 could not be raised in the PTAB proceeding, it is an issue before the district court in Delaware.

In rejecting Corning's testing, the PTAB held that the requirement in the Sufficient Adhesion Limitation of "in the presence of moisture" can only be satisfied by immersing the test samples in "liquid water." A7. Because Corning relied on test results after exposure to 95% relative humidity (the wet adhesion test from the specification), but did not immerse its test samples in liquid water, the PTAB rejected Corning's evidence as not probative of adhesion in the presence of liquid water. In construing "moisture" to mean "liquid water," the PTAB stated that it did not need to construe any other parts of the Sufficient Adhesion Limitation, including, surprisingly, the length of time or temperature for the test. The PTAB's construction was particularly surprising since DSM filed a non-contingent Motion to Amend, effectively replacing independent claims 1 and 10 with two narrower independent claims that recited a particular test (*i.e.*, the water soak delamination test) involving immersing a coating in liquid water. DSM specifically stated at oral argument that it was "conceding" claims 1 and 10. A862:10.

This Appeal thus arises from a PTAB Final Decision construing the Sufficient Adhesion Limitation in a way that excludes a test explicitly disclosed in the '666 Patent as being suitable for testing adhesion. Such an interpretation cannot be the broadest reasonable interpretation. DSM knew this and thus made a non-contingent motion to replace independent claims 1 and 10 of the '666 Patent

with claims explicitly calling for use of the water soak delamination test. A526-527. In this Appeal, DSM now finds itself having to defend claims which it already gave up on.

In failing to construe the claims in a manner consistent with DSM's concession, the PTAB's limiting construction of the term "moisture" to be "liquid water" does not comport with the plain meaning of the term "moisture," is not supported by the evidence, and is not the broadest reasonable construction of that term and the Sufficient Adhesion Limitation.

As discussed more fully below, given the indefinite nature and breadth of the Sufficient Adhesion Limitation and the necessity of applying the broadest reasonable interpretation to the claim, Corning's interpretation of the Sufficient Adhesion Limitation and use of the wet adhesion test from the specification is both reasonable and consistent with the claims and specification of the '666 Patent. It was, therefore, reversible error to reject Corning's claim interpretation and testing in favor of an unduly narrow, distorted, and unsupported claim interpretation, which both excludes from the ordinary understanding of "moisture" all forms of moisture (*e.g.*, humidity, water vapor, etc.) other than liquid water, and leaves completely undefined and indeterminate the required conditions (*e.g.*, water conditions, period of soaking time, etc.) for exposure to the liquid water.

## B. Procedural History

On November 15, 2012, Corning filed its Petition, requesting IPR with respect to all claims (claims 1–20) of the '666 Patent. A139 at 3:9-12. Four prior art references—Szum, Shustack, Coady, and Jackson—were identified in the Petition as overcoming any supposed deficiencies of the prior art evaluated during original prosecution of the application which issued as the '666 Patent. A139-140 at 3:15-4:2.

In particular, Corning challenged the claims as unpatentable under 35 U.S.C. §§ 102 (anticipation) and/or 103 (obviousness) based on the following specific grounds:

Reference[s]	Basis	Claims challenged
Szum	§§ 102 or 103	10-20
Szum and Coady	§ 103	1-9
Szum and Shustack	§ 103	1-9
Shustack	§§ 102 or 103	1, 2, 8, 10, 11, 16, 17, and 19
Shustack and Jackson	§ 103	3, 12, 18, and 20
Coady	§ 103	1, 2, and 8
Coady and Jackson	§ 103	3

A141-142 at 5:15-6:6.

DSM filed a Preliminary Response on February 21, 2013. In doing so, DSM did not provide any claim construction position. DSM stated that “the Board’s determination of the appropriate scope of these claim terms is not necessary” and offered a blanket objection to Corning’s proposed claim constructions to the extent that they did not correspond to the broadest reasonable interpretation. A262.

On May 13, 2013, the PTAB granted Corning’s Petition with regard to claims 1–20 of the ’666 Patent based on anticipation and obviousness grounds, relying on Szum, Shustack, and Jackson. A270.<sup>2</sup> The PTAB also made no explicit claim construction in the Decision Instituting IPR. A273-274.

On August 27, 2013, after institution of this IPR, DSM filed its PO Response, in which, for the first time, DSM proposed a construction of the Sufficient Adhesion Limitation. A498. DSM also filed a non-contingent Motion to Amend. On November 27, 2013, Corning filed a reply to the PO Response and an opposition to DSM’s Motion to Amend. The PTAB issued its Final Decision on May 9, 2014, in which it largely adopted DSM’s new construction of the Sufficient Adhesion Limitation, denied Corning’s request to cancel claims 1–20, and denied DSM’s Motion to Amend the claims without prejudice. A19; A22.

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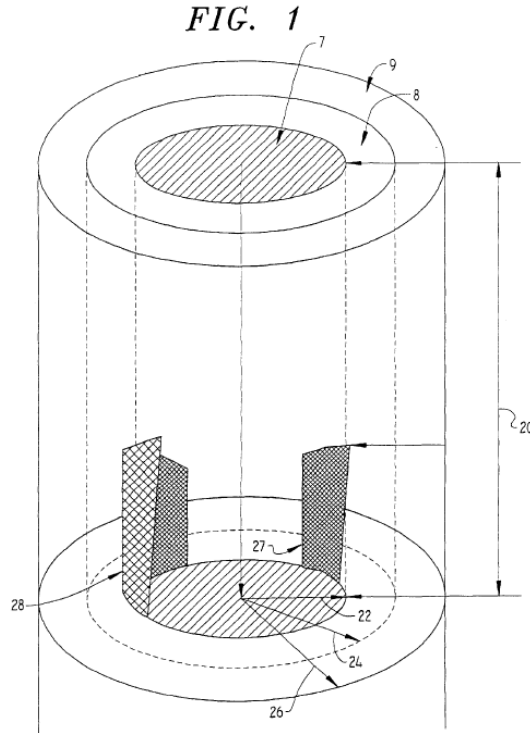
<sup>2</sup> The PTAB declined to institute an IPR based on any grounds relying on Coady, holding that these grounds were redundant in light of its decision finding that there was a reasonable likelihood that these same claims were unpatentable over several other proposed grounds. A288-289.

## **C. Statement of the Facts**

### **1. The '666 Patent**

The '666 Patent relates generally to, *inter alia*, radiation-curable inner and outer primary optical glass fiber coating compositions, coated optical glass fibers, and optical glass fiber assemblies with improved ribbon stripping capabilities. A37 at 1:22-27. The '666 Patent has two independent claims (claims 1 and 10) and 18 dependent claims, directed to various embodiments of a coating composition, a coated optical fiber, and a ribbon assembly. A70-71. The coated optical fiber described in the '666 Patent includes two coating compositions that may be applied on an optical fiber 7: the inner primary coating 8 that contacts the glass surface (A26 at Fig. 1 and A41 at 10:10-12) and the outer primary coating 9 that overlays the inner primary coating (A26 at Fig. 1 and A41 at 10:10-12). To create a cable or ribbon assembly, used in the construction of multi-channel transmission cables, a plurality of coated optical fibers are bonded together in a matrix material. A37 at 1:43-51.

Figure 1 from the '666 Patent, depicting such a coated optical glass fiber, is reproduced below:



A26. Figure 1 is a longitudinal cross-sectional view of optical glass fiber 7 coated with inner primary coating 8 and commercially available outer primary coating 9.

A40-41 at 8:11-12; 10:10-12.

The two independent claims in the '666 Patent are reproduced below:

1. A composition for coating an optical fiber, said composition comprising propoxylated nonyl phenol acrylate and an oligomer having at least one functional group capable of polymerizing under the influence of radiation, said composition after radiation cure having the combination of properties of:
  - (a) a fiber pull-out friction of less than 40 g/mm at 90° C.;
  - (b) a crack propagation of greater than 1.0 mm at 90° C.;
  - (c) a glass transition temperature of below 10° C.; and
  - (d) sufficient adhesion to said glass fiber to prevent delamination in the presence of moisture and during handling.
  
10. A composition for coating an optical fiber, said composition comprising an oligomer having an aliphatic diisocyanate residue and at least one functional group capable of polymerizing under the

influence of radiation, said composition after radiation cure having the combination of properties of:

- (a) a fiber pull-out friction of less than 40 g/mm at 90° C.;
- (b) a crack propagation of greater than 1.0 mm at 90° C.;
- (c) a glass transition temperature of -20° C. or less; and
- (d) sufficient adhesion to said glass fiber to prevent delamination in the presence of moisture and during handling.

A70.

Thus, independent claims 1 and 10 recite a coating composition for coating an optical fiber having a combination of four properties, one of which is the Sufficient Adhesion Limitation.

## **2. Prosecution of the '666 Patent**

U.S. Patent Application Serial No. 09/838,140 (“the ’140 application”), which issued as the ’666 Patent, was filed on April 20, 2001. A24. The ’140 application was filed with a preliminary amendment canceling the original claim set and adding 20 new claims directed to a composition for coating an optical fiber, a coated optical fiber having an inner primary coating and an outer primary coating, and a ribbon assembly. A144.

No rejections of any sort were ever made in the ’140 application. A144. Instead, on August 22, 2001, all claims were allowed in the first office action on the merits. A144. The examiner stated that the prior art did not disclose a



radiation-curable coating composition for a glass optical fiber with the combination of all four of the properties in the claims.<sup>3</sup> A144.

### **3. The Parties' Claim Construction Positions**

#### **a. Corning's Petition for IPR**

Corning's Petition sets forth claim construction positions with regard to several claim terms, including the Sufficient Adhesion Limitation. A151. Corning explained that the Sufficient Adhesion Limitation is not defined in the '666 Patent; however, adhesion force values of at least 5 grams/inch and at least 12 grams/inch were provided in the original claim set in the '140 application; and a preferred value for "wet adhesion" of at least about 5 grams/inch is disclosed in the '666 Patent at col. 29, lines 24-25. A51; A151. The term "wet adhesion" is described in the '666 Patent at col. 28, lines 53-58 as adhesion at 95% relative humidity (A50; A151), and the wet adhesion test procedures are described in the '666 Patent at col. 28, line 57 through col. 29, line 25 (A50-51). Accordingly, Corning used this language as guidance regarding the meaning of the Sufficient Adhesion Limitation. *See, e.g.*, A154 at 18:11-14; A162 at 26:5-8; A169 at 33:6-9; A176 at 40:3-6; A181-182 at 45:17-46:2.

Corning's expert, Dr. Michael Winningham, described the Sufficient Adhesion Limitation as follows: adhesion to glass is a property that measures the

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<sup>3</sup> In addition, no prior art rejections were made in any of the priority applications. A144.

ability of a coating to adhere to the glass of the optical glass fiber upon exposure to moisture and during handling. A999 at ¶ 71. Under certain conditions, moisture will absorb and diffuse into coatings and may build up along the glass fiber, which naturally erodes the adhesive bond. A999-1000 at ¶ 71. Similarly, the coating will undergo stresses during handling, such as in connection with cabling. A1000 at ¶ 71. It is desirable for the inner primary coating to sufficiently adhere to the glass even under wet or high-moisture conditions or during handling. A1000 at ¶ 71.

Dr. Winningham explained that there are a number of accepted methods for measuring the adhesion of a coating to glass. A1000 at ¶ 72. The test methodology section of the '666 Patent describes one such testing methodology for determining the adhesion to glass at col. 28, line 57 to col. 29, line 25, the wet adhesion method. A50-51; A1000 at ¶ 72.

Dr. Winningham explained that the only guidance with regard to what constitutes "sufficient adhesion" as set forth in the claims of the '666 Patent is that it be at least 5 grams/inch. *See, e.g.*, A1007 at ¶ 88; A1017 at ¶ 100; A1029 at ¶ 111; A1039 at ¶ 124; A1053 at ¶ 134. Dr. Winningham also confirmed that the coatings of Szum, Shustack, and Coady had adhesion to glass values that, when tested using the wet adhesion methodology described in the '666 Patent, would provide sufficient adhesion to the glass fiber to prevent delamination in the presence of moisture and during handling, in that the coatings of Szum, Shustack,

and Coady all far exceeded 5 grams/inch in the wet adhesion test. *See, e.g.*, A1007 at ¶ 88; A1017-1018 at ¶ 100; A1029 at ¶ 111; A1039-1040 at ¶ 124; A1053 at ¶ 134.

This testimony by Dr. Winningham is consistent with his later testimony explaining how moisture weakens the bonds between a coating and underlying glass, and why this is relevant to both delamination and lower results in the wet adhesion test. A2105 at ¶ 19. This is also consistent with statements in the specification of the '666 Patent. *See* A37 at 2:46-53 (“[A]ny reduction in the adhesion between the inner primary coating and the optical glass fiber increases the possibility of such undesirable delamination, especially in the presence of moisture.”).<sup>4</sup>

**b. DSM’s Patent Owner’s Response and Motion to Amend**

In its PO Response, DSM argued that the Sufficient Adhesion Limitation requires, at least, a showing that a coating does not delaminate during a 24-hour water soak delamination test, which was one of the three tests described in the specification of the '666 Patent (the other two being the wet adhesion test and the dry adhesion test). A498. Further, DSM asserted that the Sufficient Adhesion

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<sup>4</sup> The data in Table 3 of the '666 Patent is entirely consistent with this statement and Dr. Winningham’s testimony. Table 3 unambiguously illustrates four samples where lower adhesion scores in the wet adhesion test correspond to earlier delamination in a water soak test. *See* A51 at 8:26-65.

Limitation is not satisfied by the wet adhesion test Corning performed (which also was from the specification). A498.

DSM also made a Motion to Amend, requesting cancellation of claims 1 and 10, substituting them, respectively, with new claims 21 and 22, which specified that the Sufficient Adhesion Limitation be determined with a water soak delamination test.<sup>5</sup> A526-527. These substitute claims were *not* proposed in the alternative to the PO Response. A526. Instead, DSM confirmed during oral hearing that “[t]he motion to amend is not contingent.” A860:1-2; *see also* A860:14-17. In fact, DSM stated that it was “conceding Claims 1 and 10.” A862:10.

### **c. Corning’s Reply**

In the Corning Reply, Corning argued that the Sufficient Adhesion Limitation broadly defines a property of the claimed coating composition without specifying a particular procedure for determining that property. A622. Therefore, any procedure for determining sufficient adhesion to prevent delamination in the presence of moisture and during handling, including at least the wet adhesion test from the specification of the ’666 Patent, will satisfy this broad claim limitation. A622-623. Moreover, Corning pointed out that there is no requirement in the

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<sup>5</sup> DSM asserted that the Sufficient Adhesion Limitation in claims 1 and 10 implicitly required use of a 24-hour 60°C water soak delamination test. A498-499. Proposed new claims 21 and 22 explicitly required these same time, temperature, and test qualifications to meet the Sufficient Adhesion Limitation. A526-527.

claims that the term “moisture” as it appears in the Sufficient Adhesion Limitation, be narrowly construed and effectively rewritten to require immersion in liquid water and exclude exposure to a moist atmosphere. A623. The claim limitation “moisture” is also not defined as liquid water in the specification of the ’666 Patent. A623. Rather, the ’666 Patent specifically refers to the presence of “moisture” in a conditioned coating sample in a wet adhesion test at col. 29, lines 5-7. A51; A624.

Corning also submitted a responsive declaration from Dr. Winningham, which addressed the applicability of the wet adhesion test from the specification in determining adhesion of a coating and prevention of delamination in the *presence of moisture*. A2105 at ¶ 19. Dr. Winningham stated that when the bonds between a coating and underlying glass fiber are weakened with moisture, the average force value in the wet adhesion test (which is the result of the test) is lower. A2105 at ¶ 19. Weakening of the bonds due to moisture can result in delamination. A2105 at ¶ 19. Thus, the wet adhesion test is directly relevant to measuring adhesion and prevention of delamination in the presence of moisture. A2105 at ¶ 19.

#### **4. The PTAB Issues Its Final Decision**

On May 9, 2014, the PTAB issued its Final Decision in which it denied Corning’s request to cancel claims 1–20 of the ’666 Patent and denied without

prejudice DSM's Motion to Amend the claims, notwithstanding DSM explicitly conceding claims 1 and 10. A22; A862 at 19:10.

The PTAB focused its analysis on a “dispositive issue” concerning the Sufficient Adhesion Limitation, required by each challenged claim. A5. The PTAB held that, under the broadest reasonable interpretation, the term “moisture” in the phrase ““in the presence of moisture,” means liquid water—that is, a condition of 100% relative humidity.” A7. In support of this holding, the PTAB stated that the written description of the '666 Patent uses the term “moisture” in a context that suggests liquid water and cited the protocols for the wet adhesion test and the elastic modulus test from the specification of the '666 Patent. A7. In addition, the PTAB held that where the written description discusses water in a vapor form, the inventors use the word “humidity” or “atmospheric moisture,” but not “moisture” alone (citing '666 Patent, at 21:54 28:55, 67; 29:5). A47; A50; A51; A7-8.

The PTAB held that Corning relied on test results after exposure to 95% relative humidity (the wet adhesion test), but not to liquid water, and that Corning did not explain how that result is probative of adhesion in the presence of liquid water. A13-14; A19. On that basis, the PTAB held that Corning failed to show by a preponderance of evidence that the prior art coatings met the Sufficient Adhesion Limitation. A13-14; A19.

Additionally, the PTAB asserted that Corning did not identify any objective evidence explaining how a wet adhesion value correlates to an ability to withstand the hydrodynamic forces that effect delamination. A14-15. In particular, the PTAB stated that it would not “infer” a relationship between wet adhesion values and water soak delamination results in the absence of persuasive evidence, such as a technical article or expert testimony, explaining some relationship between those test results. A16. The PTAB also decided not to accept Corning’s argument that the ’666 Patent itself demonstrated a correlation between wet adhesion and an ability to withstand the hydrodynamic forces that effect delamination. A15-16. Thus, the PTAB held that Corning identified no persuasive evidence from which it could discern that the wet adhesion test evaluates for “delamination.” A17.

## SUMMARY OF THE ARGUMENT

In construing claim terms, the PTAB is required to apply the broadest reasonable interpretation “in light of the specification of the patent in which they appear.” Final Decision at 6. The Sufficient Adhesion Limitation requires that a coating have “sufficient adhesion” to glass to “prevent delamination” in the “presence of moisture” and “during handling.” The specification of the ’666 Patent describes three potentially relevant tests—the water soak delamination test, the dry adhesion test, and the wet adhesion test. Instead of applying the broadest reasonable interpretation to that limitation, and accepting that Corning’s use of one of those three tests (the wet adhesion test) was sufficient, the PTAB interpreted the phrase, in the Sufficient Adhesion Limitation, of “in the presence of moisture” to be limited to immersion in “liquid water,” and to exclude all other forms of exposure to moisture. That construction improperly narrowed the scope of the claims by excluding the two other potentially relevant test methods described in the specification. In addition, concluding that “moisture” must be “liquid water—that is, a condition of 100% relative humidity” distorts the plain meaning of the word “moisture” and is unsupported by evidence.

Reinterpreting “moisture” as “liquid water” is both incorrect and not the broadest reasonable interpretation of the Sufficient Adhesion Limitation. The PTAB’s reinterpretation has no support in the claims or the specification of the



'666 Patent. The plain meaning of the word “moisture” does not mean immersion in “liquid water,” but instead is entirely consistent with the wet adhesion test, which is carried out in a very humid environment (*i.e.*, an environment with a high level of moisture in it). Furthermore, the specification undercuts the PTAB’s construction in that the unmodified term “moisture” is used in a number of instances in the specification that unmistakably are not limited to “liquid water,” but something more extensive, including a 95% humidity environment, as in the wet adhesion test Corning performed.

Moreover, the PTAB’s equating 100% humidity with liquid water is scientifically unsound and not supported by evidence. The phenomenon that water may condense on surfaces at 100% humidity does not equate to the presence of a liquid water bath in which the surface is immersed.

The PTAB also erred in determining that the term “delamination” is only relevant to a situation in which the coating is exposed to liquid water. Again, nothing in the claims or the specification of the '666 Patent supports the PTAB’s construction. In the specification, the term “delamination” is used in a number of ways in which it is clear that the delamination concerns described had nothing to do with liquid water.

Finally, the PTAB erred in not permitting DSM to cancel claims 1 and 10 of the '666 Patent pursuant to DSM’s non-contingent motion to amend. In other

proceedings, the PTAB has consistently cancelled the claims in non-contingent motions to amend. The PTAB's refusal to do so is particularly inappropriate here, where DSM stated at oral argument that it was "conceding" claims 1 and 10. At the very least, DSM's actions with respect to its motion to amend the claims demonstrate that the PTAB's claim interpretation was erroneous and that DSM knew it.

## **ARGUMENT**

### **A. Standard of Review**

The PTAB's legal conclusions, including the PTAB's construction of claim language, are reviewed *de novo*; its factual determinations are reviewed for substantial evidence. *C.W. Zumbiel Co. v. Kappos*, 702 F.3d 1371, 1381 (Fed. Cir. 2012); *In re Abbott Diabetes Care, Inc.*, 696 F.3d 1142, 1148 (Fed. Cir. 2012). Substantial evidence is “such relevant evidence as a reasonable mind might accept as adequate to support a conclusion.” *In re Abbott Diabetes Care, Inc.*, 696 F.3d at 1148 (quoting *Consol. Edison Co. v. NLRB*, 305 U.S. 197, 229 (1938)). Applying that standard, the Court should reject the PTAB's interpretation of the Sufficient Adhesion Limitation, required in all claims, reverse the PTAB's Final Decision, and remand for cancellation of claims 1–20 of the '666 Patent.

### **B. The PTAB is Required to Apply the Broadest Reasonable Interpretation to Claim Terms in IPRs**

Pursuant to 37 C.F.R. § 42.100(b), the PTAB's own rules require it to give claims the broadest reasonable interpretation. *See also In re Abbott Diabetes Care, Inc.*, 696 F.3d at 1148. The words of claims should be “interpreted according to their broadest reasonable construction in light of the specification of the patent in which they appear.” A6; *see also In re Morris*, 127 F.3d 1048, 1054-1055 (Fed. Cir. 1997) (claims should be given “the broadest reasonable meaning of the words in their ordinary usage as they would be understood by one of ordinary skill in the

art, taking into account whatever enlightenment by way of definitions or otherwise that may be afforded by the written description contained in the [ ] specification.”).

Although an inventor may “define the specific terms used to describe his or her invention, this must be done with reasonable clarity, deliberateness, and precision.” *In re Paulsen*, 30 F.3d 1475, 1480 (Fed. Cir. 1994). In the absence of such a definition, however, limitations are not to be read into the claims from the specification. *See In re Van Geuns*, 988 F.2d 1181, 1184 (Fed. Cir. 1993).

Additionally, the PTAB may rely on testimony from persons of skill in the art “to establish that a particular term in the patent or the prior art has a particular meaning in the pertinent field.” *Phillips v. AWH Corp.*, 415 F.3d 1303, 1318 (Fed. Cir. 2005) (en banc). Expert testimony, however, may not result in a claim construction contrary to the claim construction mandated by intrinsic evidence. *Id.* Thus, while this Court defers to the PTAB’s findings concerning the credibility of expert witnesses, *see Yorkey v. Diab*, 601 F.3d 1279, 1284 (Fed. Cir. 2010), the PTAB’s reliance on expert testimony cannot result in a claim construction that varies from a construction dictated by intrinsic evidence.

**C. Corning’s Interpretation of the Sufficient Adhesion Limitation Was Reasonable, Comported with the Claims and Specification, and Was Proper under the Broadest Reasonable Interpretation**

Corning presented evidence that a prior art coating with a sufficiently high adhesion to glass when tested pursuant to the wet adhesion test in the ’666 Patent

“would have sufficient adhesion to the glass fiber to prevent delamination in the presence of moisture and during handling.” A155, citing A1007 at ¶ 88.

While the water soak delamination test may also relate to the adhesion of a coating to glass in the presence of moisture, it does not involve any handling. *See* A50 at 27:32-47 (after placement in the hot water bath, coated slides are only observed without need for further handling). The dry adhesion test involves exposing the film to 50% relative humidity followed by handling of the film using a 180 degree peel-back test; the *wet* adhesion test involves exposing the film to 95% *relative humidity* followed by handling of the film using a 180 degree peel-back test. *See* A50-51 at 28:28-29:25. Thus, both of these adhesion tests involve handling of the coating.

Significantly, the wet adhesion test is the only one of the three disclosed adhesion tests to specifically determine “adhesion” after exposure to what the ’666 Patent describes as “moisture” (which are terms found in the Sufficient Adhesion Limitation) as follows:

[Samples] were then conditioned at a temperature of 23+/-2 C. and a relative humidity of 50+/-5% for a time period of 7 days...the film to be tested for wet *adhesion* was further conditioned at a temperature of 23+/-2 C. and a relative humidity of 95% for a time period of 24 hours. A layer of polyethylene wax/water slurry was applied to the surface of the further conditioned film *to retain moisture*.

A50-51 at 28:66-29:7.

Corning's position is supported by the testimony of Dr. Winningham. In particular, Dr. Winningham testified that when the bonds between a coating and underlying glass fiber are weakened with moisture, the average force value in the wet adhesion test (which is the result of the test) is lower. A2105 at ¶ 19.

Additionally, as Dr. Winningham testified, weakening of the bonds due to moisture can result in delamination. A2105 at ¶ 19. Thus, the wet adhesion test is directly relevant to measuring adhesion and prevention of delamination in the presence of moisture. A2105 at ¶ 19.

Dr. Winningham's opinions (and Corning's claim construction) are entirely consistent with statements and test data in the '666 Patent. *See* A37 at 2:46-53 (“[A]ny reduction in the adhesion between the inner primary coating and the optical glass fiber increases the possibility of such undesirable delamination, especially in the presence of moisture.”); *see also* A51 at Table 3 (demonstrating a correlation between higher wet adhesion test results and coating compositions that pass the water soak test, and lower wet adhesion test results and coating compositions that fail the water soak test).

The PTAB refused to consider the data appearing in Table 3 of the '666 Patent (A16), in spite of the fact that Dr. Carl Taylor, DSM's expert, relied on Table 3 in his correlation of wet adhesion test results with water soak test results. A3672-3673 at ¶ 92. In particular, Dr. Taylor compared the wet adhesion results

of Szum 5B (one of the prior art coatings on which Corning relied) as reported in Szum (23 grams/inch) versus Comp. Ex. B-3 listed in Table 3 of the '666 Patent (20 grams/inch), asserting that they are “comparable,” and noted that Comp. Ex. B-3 was partially delaminated after 24 hours. A3672-3673 at ¶ 92. The inference Dr. Taylor draws is that Szum 5B would also be expected to partially delaminate after 24 hours given its comparable behavior in the wet adhesion test.<sup>6</sup>

This comparison supports Corning’s position regarding the correlation between the wet adhesion test and the water soak delamination test. Additionally, Dr. Taylor’s comparison supports Corning’s ultimate conclusion. The Sufficient Adhesion Limitation does not impose any time constraint for determining delamination, and the PTAB even acknowledged that it did not construe that limitation to require such a time constraint. A8. Dr. Taylor notes that Comp. Ex. B-3 did not exhibit any delamination after 8 hours (A51 at Table 3); hence, by Dr. Taylor’s very same logic, Szum 5B also would have been expected to display a “comparable” behavior in the water soak test—exhibiting no delamination after 8 hours.

Thus, there is more than sufficient evidence of record correlating the wet adhesion test and the water soak test, including testimony of both experts. A999-

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<sup>6</sup> With Dr. Taylor’s comments on Table 3 already before it, the PTAB’s refusal to consider Corning’s arguments regarding that table was entirely improper, particularly when the PTAB scoured the record for references to “moisture” and “delamination” that were not otherwise relied on by either party.

1000; A2105 at ¶ 19; A3672-3673 at ¶ 92. There is also unambiguous language in the '666 Patent describing the inverse relationship between adhesion to glass and delamination (*i.e.*, as adhesion to glass drops, the possibility of delamination increases). A37 at 2:46-53.

The Sufficient Adhesion Limitation broadly defines a property of the coating composition without specifying a procedure for determining that property. The only test procedure in the specification of the '666 Patent for determining adhesion that involves “moisture” and “handling” (both of which are specific requirements in the Sufficient Adhesion Limitation) is the wet adhesion test. Corning’s testing was consistent with the broadest reasonable interpretation of the Sufficient Adhesion Limitation, and reasonable and appropriate under the circumstances.

As discussed *infra*, instead of accepting Corning’s testing, the PTAB improperly construed the Sufficient Adhesion Limitation in an incorrect and narrower manner. The PTAB construction was inconsistent with the broadest reasonable interpretation, inconsistent with the plain meaning of the words used, and inconsistent with the very evidence relied upon by the PTAB. As a consequence of its improper construction of the Sufficient Adhesion Limitation,



the PTAB wholly discounted the evidence supporting Corning's Petition.<sup>7</sup> That is reversible error.

**D. The PTAB Did Not Properly Apply the Broadest Reasonable Interpretation to the Sufficient Adhesion Limitation**

The PTAB determined that Corning did not meet its burden for two reasons. First, the PTAB held that the “broadest reasonable interpretation of the term ‘moisture’ is liquid water—that is, a condition of 100% relative humidity.” A7. Having so construed the term “moisture” as used in the Sufficient Adhesion Limitation, the PTAB found “[a] dispositive question thus arises: Does Corning show by a preponderance of evidence that the Szum coating exhibits sufficient adhesion to prevent delamination from glass in the presence of liquid water?” A10. Since Corning did not expose the prior art samples to “liquid water,” the PTAB determined that Corning had not satisfied its burden of proof. A14.

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<sup>7</sup> Even if the broadest reasonable interpretation was not the proper standard, which it clearly is, reading limitations into the claims is not proper in patent actions. *See, e.g., N. Am. Container, Inc. v. Plastipak Packaging, Inc.*, 415 F.3d 1335, 1348 (Fed. Cir. 2005) (“[U]nless required by the specification, limitations that do not otherwise appear in the claims should not be imported into the claims.”); *E.I. du Pont de Nemours & Co. v. Phillips Petroleum Co.*, 849 F.2d 1430, 1433 (Fed. Cir. 1988) (“It is entirely proper to use the specification to interpret what the patentee meant by a word or phrase in the claim. But this is not to be confused with adding an extraneous limitation appearing in the specification, which is improper. By ‘extraneous,’ we mean a limitation read into a claim from the specification wholly apart from any need to interpret what the patentee meant by particular words or phrases in the claim. Where a specification does not require a limitation, that limitation should not be read from the specification into the claims.”) (citations and quotations omitted) (emphasis omitted).

Second, as a purported “independent basis” for its conclusion, the PTAB determined that Corning had presented insufficient evidence to “support an inference,” A14, that the “wet adhesion test evaluates for ‘delamination.’” A17. Both findings are fatally flawed, because they rely on an interpretation of the Sufficient Adhesion Limitation that is both incorrect and narrower than appropriate under the broadest reasonable interpretation standard, ignore the plain meaning of the word “moisture,” are unsupported by the evidence cited by the PTAB, and lack a factual basis founded in the intrinsic evidence of the ’666 Patent.

Claims 1 and 10 are silent with respect to the proper way to determine what meets the Sufficient Adhesion Limitation, and what is meant by the specific term “moisture” as used in the Sufficient Adhesion Limitation. Likewise, while the specification describes a number of tests, there is nothing in the specification that identifies with clarity, deliberateness, and precision which if any of the described tests, in whole or in part, should be used to determine what meets that limitation or what is meant by the term “moisture.” Nor does the specification explain with clarity, deliberateness, and precision what results constitute “sufficient adhesion” in accordance with the Sufficient Adhesion Limitation, or what it means to “prevent delamination,” making the Sufficient Adhesion Limitation particularly unclear and unduly broad.

This is in direct contrast to the three other material property limitations in independent claims 1 and 10 of the '666 Patent. Those claims recite specific, numerical ranges for fiber pull-out friction, crack propagation, and glass transition temperature, and there is one test description in the specification for each of those properties. *See* A52 at 31:44-59 (fiber pull-out friction); A52 at 31:16-29 (crack propagation); and A53-54 at 34:66-35:31 (glass transition temperature). DSM chose to claim the fiber pull-out friction, crack propagation, and glass transition temperature properties using numerical ranges and using claim language that corresponds to test descriptions in the specification; however, DSM chose not to do so with the Sufficient Adhesion Limitation. DSM should not now be allowed to selectively narrow the interpretation of the Sufficient Adhesion Limitation to escape an unpatentability ruling.

Central to its position was DSM's new claim construction of the term "moisture" in the Sufficient Adhesion Limitation – a claim construction DSM did not advance in its Preliminary Response. DSM argued that "'in the presence of moisture' means exposure to liquid water—that is, 100% relative humidity—as would be present, for example, in the water soak delamination test that is described in the '666 patent." A7; A506-507. Further, DSM argued that the wet adhesion test Corning conducted "does not evaluate for delamination, which is caused by exposure to liquid water." A9; A498-499; A3650-3654 at ¶¶ 46-53.

Though the PTAB indicated that it was giving the Sufficient Adhesion Limitation the broadest reasonable interpretation (A6), the PTAB adopted DSM's new claim construction argument and imported into the Sufficient Adhesion Limitation an incorrect and narrowing feature found nowhere in the claim—interpreting the term “moisture” to mean immersion in “liquid water.” This narrowing importation is not supported by the specification; in fact, it is contrary to the specification.

By choosing an incorrect and narrower interpretation of the Sufficient Adhesion Limitation over a broader, reasonable alternative, the PTAB failed to follow its own rules and committed reversible legal error. *See Phillips*, 415 F.3d at 1318 (expert testimony may not result in a claim construction contrary to the claim construction mandated by intrinsic evidence); *Tempo Lighting, Inc. v. Tivoli, LLC*, 742 F.3d 973, 977-78 (Fed. Cir. 2014) (upholding PTAB's rejection of the examiner's narrow claim interpretation where it was contrary to intrinsic evidence and added “limitations that lack support in any form of intrinsic evidence”); *Leo Pharm. Prods., Ltd. v. Rea*, 726 F.3d 1346, 1352-53 (Fed. Cir. 2013) (PTAB “erred by narrowing the definition of ‘storage stable’ to something far short of its broadest reasonable meaning”); *Cisco Sys., Inc. v. Lee*, 557 Fed. Appx. 963, 974-75 (Fed. Cir. 2014), *cert. denied*, No. 14-221, 2014 WL 4206949 (Oct. 14, 2014) (reversing

PTAB based on finding that the PTAB narrowed the claim term by reading in an “improper limitation of the claimed multiplexer”).

**1. Nothing in the ’666 Patent Supports the PTAB’s Interpretation of “Moisture” as “Liquid Water”**

There is no requirement in the claims that the term “moisture” in the Sufficient Adhesion Limitation involves immersion in or exposure to liquid water rather than an atmosphere containing a high level of moisture (*e.g.*, 95% humidity). Nor is the term “moisture” in the Sufficient Adhesion Limitation defined as liquid water in the specification. As discussed *infra*, the intrinsic evidence demonstrates that the patentee used the term “moisture” in a broad fashion, encompassing a wide range of moist environments without being limited to only meaning “liquid water.” By changing the actual language of the Sufficient Adhesion Limitation from “moisture” to immersion in “liquid water,” the PTAB critically and improperly rewrote and *narrowed* the claims, and thus failed to apply the broadest reasonable interpretation. *See, e.g., Facebook, Inc. v. Pragmaus AV, LLC*, \_\_ Fed. Appx. \_\_, Nos. 2013-1350, 2013-1351, 2014 WL 4454956, at \*2 (Fed. Cir. Sept. 11, 2014) (reversing PTAB’s claim construction that “unduly narrows the claim term . . . because nothing in the plain language of the claims or in the specification or prosecution history requires such a limitation.”).

In fact, the use of the term “moisture” in the claims and specification of the ’666 Patent demonstrates that the term “moisture” is not limited to only meaning

“liquid water,” and certainly not immersion in liquid water. Rather, as noted *infra*, the specification makes clear that the term “moisture” at least encompasses 95% humidity, making the wet adhesion test Corning conducted a reasonable and acceptable method of determining whether a coating had “sufficient adhesion” in the presence of “moisture” to satisfy the Sufficient Adhesion Limitation.

The patentee did not define “moisture”; nor did the patentee in any way disclaim the full scope of the term “moisture.” Accordingly, there is no reason to depart from the plain and customary meaning of the term. *See, e.g., Facebook*, 2014 WL 4454956 (reversing the PTAB’s claim construction in an *inter partes* reexamination for failure to apply the broadest reasonable interpretation, where the patentee did not define the term at issue and did not disclaim the full scope of the term, yet the PTAB applied an interpretation more narrow than the broadest reasonable interpretation); *see also In re Shoner*, 341 Fed. Appx. 642, 646 (Fed. Cir. 2009) (“The PTO is required to give claim terms their ‘broadest reasonable interpretation consistent with the specification,’ and there is nothing in the specification that would explicitly limit the air chamber to a particular structure.”).

The PTAB’s conclusion that “moisture” must be “liquid water” distorts the plain meaning of the word “moisture.” Inclusion of a wet adhesion test (*i.e.*, 95% relative humidity test results) within the meaning of the Sufficient Adhesion Limitation is entirely consistent with the plain meaning of the words “humidity”

and “moisture.” This is demonstrated by the reference to “moisture” in the dictionary definition of “humidity” as “a moderate degree of wetness (as of a solid surface or the air) perceptible to the eye or to touch; *moisture*, dampness.” *See, e.g., Webster’s Third New International Dictionary of the English Language Unabridged*, (2002), p. 1101.<sup>8</sup> Certainly, the wet adhesion test’s use of a very humid environment has a moisture level encompassed by the moisture language in the Sufficient Adhesion Limitation.

In addition, the plain meaning of “moisture” is “liquid (as water) diffused or condensed in relatively small quantity and dispersed through a gas as invisible vapor or as fog or in or on a solid body in insensible form or as a sensible dampness or condensed on a cool surface as visible dew.” *See, e.g., Webster’s Third New International Dictionary of the English Language Unabridged*, (2002), pp. 1453-54. That definition of “moisture” demonstrates that “moisture” is not commonly understood to mean *only* “liquid water.” “Liquid diffused” and liquid “dispersed through a gas” are not immersion in “liquid water”; instead, “liquid diffused” or liquid “dispersed through a gas” comport with Corning’s exposing its

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<sup>8</sup> Consideration of dictionary definitions here is entirely proper. *See Phillips*, 415 F.3d at 1322-23 (quoting *Vitronics Corp. v. Conceptronic, Inc.*, 90 F.3d 1576, 1584 n. 6 (Fed. Cir. 1996)) (“judges are free to consult dictionaries and technical treatises ‘at any time in order to better understand the underlying technology and may also rely on dictionary definitions when construing claim terms, so long as the dictionary definition does not contradict any definition found in or ascertained by a reading of the patent documents.’”).

test samples to 95% humidity.

From the dictionary definitions of “humidity” and “moisture,” it is clear that Corning’s use of the wet adhesion test to prove that the prior art met the Sufficient Adhesion Limitation was entirely proper. On the other hand, the PTAB’s conclusion that “moisture” means “liquid water” is not supported by the definitions of “moisture” and “humidity,” but instead distorts the plain meaning of those words beyond recognition.

The PTAB’s equating 100% relative humidity with liquid water (A7 and A14), is particularly inappropriate since it is unsupported by the evidence of record and is scientifically incorrect. The PTAB seems to rely on Dr. Taylor’s testimony for this conclusion, but Dr. Taylor says nothing of the kind. Instead, as the PTAB notes (A7), Dr. Taylor simply states that on a surface, “moisture condenses at 100% relative humidity.” A3651 at ¶ 48. However, the formulation of droplets of water on a surface by condensation does not equate to immersing that surface in a liquid water bath. No scientist would equate these concepts, and certainly Dr. Taylor does not. Accordingly, the PTAB’s conclusions regarding the relationship between 100% relative humidity and liquid water are unsupported by the evidence of record or by scientific principles.

To support its finding that “moisture” was limited to “liquid water,” the PTAB primarily pointed to two test descriptions in the specification where the term



“moisture” was used, according to the PTAB, “in a context that suggests liquid water.” A7. Neither of those test descriptions, however, nor any other uses of the term “moisture” in the ’666 Patent (whether cited by the PTAB or not), supports the PTAB’s change of the commonly understood term “moisture” to “liquid water.” It does just the opposite. Both tests use the term “moisture” in a way that it is at least broad enough to encompass 95% humidity as used in the wet adhesion test.

**a. The Wet Adhesion Test Does Not Support the PTAB’s Change of “Moisture” to “Liquid Water”**

The PTAB cited the procedures of the wet adhesion test (the test Corning conducted) as supporting the PTAB’s interpretation of moisture as meaning liquid water. A7. Yet, the reverse is true.

As an initial matter, the name of the test is suggestive—the “wet” adhesion test. A50 at 28:54. More importantly, the wet adhesion test provides that film samples cured on glass microscope slides should be conditioned “at a temperature of 23+/-2° C. and a relative humidity of 95% for a time period of 24 hours.” Thereafter, “[a] layer of polyethylene wax/water slurry [is] applied to the surface of the further conditioned film *to retain moisture*.” A50-51 at 28:66-29:7.

While the PTAB cites this language as supportive of an interpretation of “moisture” as “liquid water,” the reverse is true. The purpose of the application of a wax/water slurry to the film is to “*retain moisture*” in the film sample after its

exposure to 95% relative humidity—not liquid water. The only “moisture” that could be “retain[ed]” by the wax/water slurry is the “moisture” resulting from the conditioning at 95% humidity.

DSM’s expert, Dr. Taylor, testified that the moisture referred to in the wet adhesion test was the “*water* molecules that [ ] diffused into the inner primary coating while conditioned at 95 percent for 24 hours.” Yet, Dr. Taylor argued that the word “moisture” as used in the wet adhesion test was somehow different than how the word “moisture” was used in the Sufficient Adhesion Limitation. A1713 at 469:15-470:18.

There is no support for Dr. Taylor’s (or the PTAB’s) conflicting interpretations of “moisture.” Absent specific evidence to the contrary, and there is none, the word “moisture,” without any additional modifiers, should be interpreted the same way across the ’666 Patent. *See Phillips*, 415 F.3d at 1314 (“claim terms are normally used consistently throughout the patent”). Thus, the PTAB should not apply one definition to “moisture” as used in the Sufficient Adhesion Limitation and another definition to “moisture” as used in the wet adhesion test.

**b. The Elastic Modulus Test Description Does Not Support the PTAB’s Change of “Moisture” to “Liquid Water”**

To support its construction, the PTAB also pointed to the elastic modulus test method detailed in the specification, wherein heat is applied to the test samples

to remove “moisture” before conducting the test. A7, citing A54 at 35:24-26.<sup>9</sup>

Yet, *no* liquid water or even high-humidity conditioning is discussed in the elastic modulus test procedures *at all*. See A53-54 at 34:65-35:31. Thus, the only “moisture” that the heating step in the test could remove prior to testing was “moisture” present as a result of *ambient humidity*.

Contrary to the PTAB’s Final Decision, the “context in which the term ‘moisture’ appears in the specification” (A8) does *not* refer to liquid water alone. In fact, both times the term “moisture” was used in the test descriptions the PTAB cites, it was used in a way that indicates a *broader* meaning than liquid water—*i.e.*, as 95% humidity in the wet adhesion test and as ambient humidity in the elastic modulus test.

**c. Other Uses of the Term “Moisture” in the ’666 Patent Undercut the PTAB’s Interpretation of that Term**

Elsewhere in the ’666 Patent, the term “moisture” is likewise used without expressing any intent to exclude ambient moisture in favor of immersion in liquid water. See A37 at 2:46-53 (“While [US Pat. 5,373,578] discloses ... that the

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<sup>9</sup> Neither party argued to the PTAB that the elastic modulus test had any relevance to the Sufficient Adhesion Limitation. As described in the ’666 Patent, the elastic modulus test is relevant to the glass transition temperature of a coating. A53-54 at 34:67-35:1. DSM did not dispute that the prior art coatings met the glass transition temperature limitations in the claims of the ’666 Patent. Thus, the PTAB independently searched the ’666 Patent and referenced this test in regard to the Sufficient Adhesion Limitation only because it uses the term “moisture” in the test description.

adhesion between the inner primary coating and the optical glass fiber should be sufficient to prevent delamination of the inner primary coating from the optical glass fiber, any reduction in the adhesion between the inner primary coating and the optical glass fiber increases the possibility of such undesirable delamination, especially in the presence of moisture.”); A49 at 25:42-47 (“The composite oligomer functions to tailor the properties of formulations which exhibit too great a coefficient of friction or too low adhesion. Specifically, the composite oligomer can increase the adhesion if the adhesion is unacceptably low, and in particular unacceptably low in the presence of moisture.”).

In one instance, the specification of the ’666 Patent separately and distinctly refers to both “water” and “atmospheric moisture,” and indicates that both can have the same effect. *See* A47 at 21:45-54 (“Generally, at least one hydrolyzable ‘-Si-O-R’ linkage will be present in the glass coupling moiety to facilitate coupling to the surface of the optical glass fiber. Preferably, there is more than one such linkage. *Hydrolyzable means that this linkage is sensitive to reaction with water to generate ‘-SiOH’ linkages. In turn, ‘-Si-OH’ linkages are believed to condense to form ‘-Si-O-Si-’ linkages. In many cases, hydrolysis may even begin to occur with exposure to atmospheric moisture.*”

The PTAB ascribes some significance to this last instance of using the term “atmospheric moisture” as opposed to simply “moisture.” A7-8. However, the

PTAB ignores the fact that the same section specifically uses the word “water” instead of “moisture,” demonstrating that DSM recognized water and atmospheric moisture to be different, and that DSM was fully able to specify water when it wanted to do so. *See Enzo Biochem, Inc. v. Applera Corp.*, 599 F.3d 1325, 1333 (Fed. Cir. 2010) (“The applicants knew how to claim a linkage group . . . , as they did in the [related] patents, but specifically omitted that language from the claims of the [patent at issue].”).

**d. One Potential Use of the Coating Does Not Dictate that “Moisture” Must Equal Water**

As purported additional support for its conversion of “moisture” to “liquid water,” the PTAB identified language in the ’666 Patent stating that “a ‘ribbon assembly *can* be buried under ground or water for long distance connections,’ ... which is consistent with the proposition that an optical fiber coating must endure long periods of immersion in liquid water without delaminating.” A8, quoting A70 at 67:20-22. As an initial matter, that bald reference, which does not even use the term “moisture,” should not limit the breadth of the claim term “moisture” absent an explicit indication or disclaimer. *See, e.g., Facebook*, \_\_ Fed. Appx. \_\_, 2014 WL 4454956.

The ’666 Patent describes a “ribbon assembly” as a “plurality of coated optical glass fibers which are bonded together in a matrix material. For example, the matrix material can encase the optical glass fibers, or the matrix material can

edge-bond the optical glass fibers together.” A37 at 1:47-51. In relying on the statement in the ’666 Patent that “a ribbon assembly can be buried,” the PTAB apparently ignored the fact that the ribbon assembly would be “bonded together in a matrix material” (A37 at 1:44-50) and, further, that the ribbon is commonly protected in cable jacketing or sheathing. *See* A960 at 1:14-18. Further, the PTAB evidently reached this conclusion without considering any evidence on whether the optical fiber itself would be immersed in liquid water or the impact of the matrix material or jacketing/sheathing on the water resistance of the ribbon assembly over “long periods of immersion in liquid water.” A8.

Moreover, given that the PTAB specifically “decline[d] to resolve what temperature, *or length of time*, of exposure to liquid water the coating must endure, without delaminating” (A8), the reference to the coating possibly experiencing “long periods of immersion in liquid water” (A8) is inappropriate.

**e. Extrinsic Evidence Does Not Support the PTAB’s Narrowed Claim Interpretation**

Resorting to extrinsic evidence further undercuts the PTAB’s claim construction and supports Corning’s construction. When Dr. Taylor was disinterested, *i.e.*, before becoming involved in this proceeding, he co-authored a paper in which optical fibers were aged for a period of time at elevated temperature and 95% relative humidity. A1715 at 476:1-477:2. In his paper, Dr. Taylor and his co-authors report that the test showed “[t]he decrease in modulus of the primary

coating after *exposure to moisture* is a result of hydrolysis of the network structure that decreases the crosslink density.” A1715 at 476:14-477:2. Although during his deposition Dr. Taylor tried to distance his opinions expressed in this IPR from the language used in his earlier paper by blaming his co-authors for the choice of language and suggesting that he had perhaps not read the paper carefully enough before publication, the fact remains that other skilled scientists—including Dr. Taylor at one point in time—recognized 95% humidity as involving exposure to “moisture.” A1714-1716 at 473:11-480:4. The conditioning at 95% humidity that Dr. Taylor and his co-authors referred to as involving “exposure to moisture” is the same 95% humidity conditioning called for in the wet adhesion test Corning performed.

**2. The PTAB’s Determination that the Term “Delamination” Should be Understood Only in Connection with Exposure to Liquid Water is Incorrect**

As a purportedly “independent basis” for its decision, the PTAB ruled that Corning had presented insufficient evidence to support an inference that the wet adhesion test evaluates for “delamination.” A17. This determination effectively adopted DSM’s argument in its PO Response that the water soak test is the only method disclosed in the ’666 Patent for assessing “delamination.” A9, citing A498. However, this argument was later abandoned by DSM and was, in fact, contradictory to the position of DSM’s expert, Dr. Taylor.

The PTAB's focus on the term "delamination" as only being applicable to the water soak test is not supported by the intrinsic evidence. While the water soak delamination test described in the '666 Patent requires soaking in water, A50 at 27:29-47, the specification elsewhere makes clear that "delamination" may occur in the presence or absence of "moisture."

For example, the '666 Patent raises the concern that some coatings are "easily peelable, as by rubbing with finger pressure, [and thus] the coating has insufficient adhesion to the surface of the optical glass fibers to prevent *delamination* during most uses." A38 at 3:53-56; *see also* A41 at 10:1-3 (appropriate to adjust adhesion "to ensure *delamination* of the inner primary coating from the surface of the optical glass fiber during ribbon stripping");<sup>10</sup> A37 at 2:50-53 ("...any reduction in the adhesion between the inner primary coating and the optical glass fiber increases the possibility of such undesirable delamination, especially in the presence of moisture.");<sup>11</sup> A46 at 19:12-14 ("If the

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<sup>10</sup> "Ribbon stripping" is removing the coating(s) from an optical fiber and does not require the presence of moisture. As described in the '666 Patent, one method uses a ribbon stripping device, which applies heat and pressure to the fiber to soften the coating and allow the coating to be stripped from the fiber. *See* A37 at 2:19-31.

<sup>11</sup> This discussion of the impact of reduced adhesion on "delamination" in the presence of "moisture" directly supports Dr. Winningham's testimony that there is a correlation between the wet adhesion test results achieved by a coating and its resistance to delamination in the water soak test. *See* A2105 at ¶ 19. Although the PTAB searched the '666 Patent for mention of "moisture" and "delamination" in preparing its Final Decision (even where those references were not cited by either



adhesion is reduced, then undesirable delamination of inner primary coating from the surface of the optical glass fiber can occur.”); A63 at 54:45-54 (“The use of slip agents may cause undesirable delamination of the inner primary coating during use of the ribbon assembly in hot and wet environments, such as tropical environments.”).

Consistent with those statements confirming the inverse relationship between adhesion to glass and susceptibility to delamination, Table 3 of the ’666 Patent illustrates this inverse relationship with four coatings that exhibit varying degrees of glass adhesion measured by the wet adhesion test (in grams/inch). The stronger the measured strength of glass adhesion, the longer the coating survived the water soak test before delaminations were observed. A51 at Table 3; A853 at 10:11-21. Further, Dr. Winningham clearly testified that such an inverse relationship between glass adhesion and delamination exists. A2105 at ¶ 19 (“[T]he wet adhesion test is directly relevant to measuring adhesion and prevention of delamination in the presence of moisture.”).

There is nothing in the claims or the specification that supports the PTAB’s assertion that “delamination” requires exposure to “liquid water.”<sup>12</sup> Indeed, the

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party), the PTAB failed to acknowledge, let alone address, such references where they undercut the PTAB’s narrowed interpretation of the relevant terms.

<sup>12</sup> The plain-language understanding of “delamination” is “separation (as of plywood) into constituent layers.” *See, e.g.*, Webster’s Third New International

end result of the wet adhesion test performed by Corning is delamination of the coating from the underlying glass following exposure of the coating to 95% humidity (hydrolytic forces) and subjecting the same to handling (mechanical forces). A870 at 27:5-8.

The multiple instances where the word “delamination” is used and liquid water or even moisture is not mentioned, demonstrate that the PTAB did not accord the Sufficient Adhesion Limitation its broadest reasonable interpretation. Rather, the term “delamination” is used broadly, and certainly in such a way that delamination concerns are raised in dry conditions and also in connection with “moisture” or humid environments, such as the “moisture” present in the wet adhesion test Corning performed. Moreover, DSM, when arguing that both a water soak delamination test and a wet adhesion test were required for the Sufficient Adhesion Limitation, acknowledged that the wet adhesion test does, in fact, determine delamination, at least as the result of “handling.”<sup>13</sup> The PTAB’s improperly narrowed interpretation of “delamination,” as used in the Sufficient

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Dictionary of the English Language Unabridged, (2002), p. 595. There is nothing in that commonly understood definition that requires exposure to “liquid water.”

<sup>13</sup> At oral argument, DSM changed its original position that the Sufficient Adhesion Limitation required “at least the water soak delamination test” and, without explaining why, argued that *both* the water soak delamination test and the wet adhesion test Corning conducted were required. A869-870; *see also* A3654 at ¶ 53 (“[t]he 60°C water soak delamination test is used to measure the resistance of the *coating-glass bonds* to hydrolysis...a peel test measures the resistance of *those bonds* to a mechanical force applied to the coating.”).

Adhesion Limitation, is not supported by the intrinsic evidence in the '666 Patent and constitutes reversible error by the PTAB.

Moreover, the PTAB's finding that the water soak test is the only valid method to measure delamination subverts the Sufficient Adhesion Limitation. The Sufficient Adhesion Limitation addresses how delamination of the coating occurs in response to *both* "exposure to *moisture* [and] *handling*." The water soak test does *not* involve any form of "*handling*"; as the name of the test states, the coated fiber is merely "soaked" in liquid. The wet adhesion test, chosen by Corning, on the other hand, addresses *both* moisture and *handling*. Corning's test, the wet adhesion test, was set forth in the specification and clearly addresses both of these criteria. It is reversible error for the PTAB to exclude this test, requiring instead another test that only addresses soaking in water.

### **3. Conclusion**

The Sufficient Adhesion Limitation broadly defines a property of the coating composition without specifying a procedure for determining that property. The procedure Corning followed, the wet adhesion test, was detailed in the specification of the '666 Patent. Through expert testimony and support from the specification of the '666 Patent, Corning demonstrated how a wet adhesion value correlates to an ability to withstand the hydrodynamic and mechanical forces that effect delamination in the presence of moisture and during handling. Thus, the wet

adhesion test is directly relevant to measuring adhesion and prevention of delamination in the presence of moisture and during handling. A2105 at ¶ 19;<sup>14</sup> *see also* A51 at Table 3.

The PTAB's only basis for its decision is the incorrect and narrowed claim construction it gave to the Sufficient Adhesion Limitation (to require exposure to liquid water) and the undisputed fact that Corning did not carry out such a test. This was not a correct claim interpretation and Corning's use of the wet adhesion test was proper. The PTAB, in granting Corning's Petition, agreed at that time that there was a reasonable likelihood that Corning would be able to establish that the claims were unpatentable (relying on Corning's wet adhesion test results). The PTAB in its Final Decision identified no other grounds for holding that Corning did not meet its burden. Therefore, if the Court agrees with Corning's interpretation of the Sufficient Adhesion Limitation, then claims 1–20 of the '666 Patent should be held to be unpatentable on the same grounds upon which the Petition was granted.

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<sup>14</sup> Oddly, the PTAB states that “Dr. Winningham repeats, verbatim, attorney argument set forth in the petition, but identifies no objective evidence explaining how a wet adhesion value ... correlates to an ability to withstand the hydrodynamic forces that effect delamination.” A14-15. Dr. Winningham was not parroting the Petition; rather, quite the opposite, the Petition cited to Dr. Winningham's testimony as the evidence on which the Petition relied. *See* A151, citing A1005 at ¶ 82.

**E. The PTAB Committed Reversible Error by Refusing to Accept DSM's Non-Contingent Request to Cancel Claims 1 and 10**

DSM filed a non-contingent motion to amend, whereby DSM cancelled claims 1 and 10 and proposed to substitute, respectively, new claims 21 and 22. A526-527; A860:1-2. DSM confirmed during oral hearing that it was “conceding Claims 1 and 10.” A862:10.<sup>15</sup> In the Final Decision, the PTAB erred in refusing to cancel claims 1 and 10 of the '666 Patent. A20.

The PTAB has made clear that a motion to amend in an IPR is not an amendment that is entered as a matter of right; instead the patent owner bears the burden of establishing that it is entitled to the relief requested. 37 C.F.R. § 42.20(c). For motions to amend, the PTAB has issued two guiding opinions: *Idle Free Sys., Inc. v. Bergstrom, Inc.*, IPR2012-00027 (PTAB, June 11, 2013) (Paper 26) (designated “Informative”); and *Int'l Flavors & Fragrances Inc. v. U.S.*, IPR2013-00124 (PTAB, May 20, 2014) (Paper 12) (designated “Informative”).

In *Idle Free*, patent owner Bergstrom did not address the unpatentability of certain claims over the cited prior art in its Patent Owner response and later conceded during a conference call that those claims were unpatentable. *Idle Free*, slip op. at 2. Based on these circumstances, the PTAB confirmed that it would cancel those claims. *Id.* The PTAB also identified the criteria to be satisfied for a

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<sup>15</sup> The PTAB acknowledged DSM's concession in its Final Decision. A20.

motion to amend. One of those criteria concerned any contingencies of the amendment. The PTAB clarified that patent owners must “adopt a claim-by-claim approach to specifying the *contingency of substitution, e.g., which claim for which claim and in what circumstance.*” *Idle Free*, slip op. at 10 (emphasis added).

In *International Flavors*, the patent owner filed a motion to amend requesting cancellation of all claims (*i.e.*, claims 1–26) and the substitution of claims 27–45. *Int’l Flavors*, slip op. at 2. The PTAB determined that the patent owner demonstrated patentability of all proposed substitute claims except for claim 45. *Id.* at 18. Consequently, the PTAB granted the patent owner’s request to cancel claims 1–26 and the patent owner’s motion to enter substitute claims 27–44, but denied the motion to enter substitute claim 45. *Id.* at 18-19.

Two principles emerge from these “informative” PTAB decisions: (i) it is the patent owner’s responsibility (as the moving party) to designate whether proposed amendments are contingent or non-contingent and the circumstances of any such contingency, *see Idle Free*, slip op. at 10; and (ii) where the moving party identifies claims to be cancelled in a non-contingent motion to amend, nothing more is required to warrant cancellation of those claims by the PTAB. *See Idle Free*, slip op. at 2; *Int’l Flavors*, slip op. at 18-19.

Consistent with its “informative decision” in *Idle Free*, the PTAB has repeatedly cancelled claims where patent owners request cancellation of claims by

making motions to amend non-contingent. *See Adidas AG v. Nike, Inc.*, IPR2013-00067 (PTAB, Apr. 28, 2014) (Paper 60), slip op. at 8-10, 41-42 (granting non-contingent request to cancel claims 1–46 and denying introduction of substitute claims 47-50); *Gnosis S.p.A. v. Merck & CIE*, IPR2013-00117 (PTAB, June 20, 2014) (Paper 71), slip op. at 2-3, 45 (granting non-contingent motion to cancel certain challenged claims); *Gnosis S.p.A. v. S. Alabama Med. Sci. Found.*, IPR2013-00118 (PTAB, June 20, 2014) (Paper 64), slip op. at 2-3, 42 (granting non-contingent motion to cancel all but one of the challenged claims); *Intelligent Bio-Systems, Inc. v. Illumina Cambridge Ltd.*, IPR2013-00128 (PTAB, July 25, 2014) (Paper 92), slip op. at 4-5, 29, 31 (granting non-contingent motion cancelling all claims and denying entry of substitute claims); *Silver Peak Sys., Inc. v. Riverbed Tech., Inc.*, IPR2014-00149 (PTAB, Aug. 7, 2014) (Paper 20), slip op. at 2, 4 (granting non-contingent motion to cancel all claims); *Silver Peak Sys., Inc. v. Riverbed Tech., Inc.*, IPR2014-00128 (PTAB, Aug. 7, 2014) (Paper 20), slip op. at 2-4 (granting non-contingent motion to cancel all claims); *Reg Synthetic Fuels, LLC v. Neste Oil Oyj*, IPR2013-00178 (PTAB, Aug. 29, 2014) (Paper 63), slip op. at 2-3, 20-21 (granting non-contingent motion to cancel all claims and denying entry of substitute claims). Even where the patent owner failed to designate its motion to amend as contingent, but later asserted that it intended to do so, the PTAB decided that the patent owner had “waived any issues with respect to claims

1–4” and granted the request to cancel claims 1–4. *Bloomberg Inc. v. Markets-Alert PTY Ltd.*, CBM2013-00005 (PTAB, Mar. 26, 2014) (Paper 71), slip op. at 11-12.

The only departure from this consistent PTAB practice of cancelling claims that the patent owner identifies for cancellation in a non-contingent motion to amend is this case. Here, DSM filed a motion to amend which included a non-contingent request to cancel claims 1 and 10. A526-527. DSM even confirmed during oral hearing that “[t]he motion to amend is not contingent.” A860:1-2. When pressed during oral hearing, DSM even clarified that if the PTAB concludes that unpatentability of claims 1 and 10 was not proven, then DSM still wanted to amend the ’666 Patent. A860:14-17. Indeed, under questioning by the PTAB, DSM confirmed that it was “conceding Claims 1 and 10.” A862:10. Despite these circumstances, the PTAB departed from its otherwise consistent practice and declined to cancel claims 1 and 10 of the ’666 Patent. This is an abuse of discretion, which this Court should not countenance. This Court should reverse the PTAB dismissal of DSM’s Motion to Amend, and order claims 1 and 10 cancelled.



## CONCLUSION

For the reasons above, the PTAB's Final Decision should be reversed or, at the least, vacated and remanded for further proceedings in light of proper claim construction.

Dated: October 21, 2014

Respectfully submitted,

By: /s/Michael L. Goldman

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# **ADDENDUM**

Final Written Decision  
(A1-A23)

U.S. Patent No. 6,339,666  
(A24-A71)

Trials@uspto.gov  
571-272-7822

Paper 92  
Entered: May 9, 2014

UNITED STATES PATENT AND TRADEMARK OFFICE

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BEFORE THE PATENT TRIAL AND APPEAL BOARD

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CORNING INCORPORATED  
Petitioner

v.

DSM IP ASSETS B.V.  
Patent Owner

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Case IPR2013-00045  
Patent 6,339,666 B2

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Before FRED E. McKELVEY, GRACE KARAFFA OBERMANN,  
JENNIFER S. BISK, SCOTT E. KAMHOLZ, and ZHENYU YANG,  
*Administrative Patent Judges.*

OBERMANN, *Administrative Patent Judge.*

FINAL WRITTEN DECISION  
*35 U.S.C. § 318(a) and 37 C.F.R. § 42.73*

Case IPR2013-00045

Patent 6,339,666 B2

## I. INTRODUCTION

A. *Background*

Petitioner, Corning Incorporated (“Corning”), filed a petition on November 15, 2012, for *inter partes* review of claims 1-20 (“the challenged claims”) of U.S. Patent No. 6,339,666 B2 (Ex. 1001 (“the ’666 Patent”)) pursuant to 35 U.S.C. §§ 311-319. Paper 2 (“Pet.”). Patent Owner, DSM IP Assets B.V. (“DSM”), filed a preliminary response on February 21, 2013. Paper 10 (“Prelim. Resp.”). On May 13, 2013, the Board instituted trial on six grounds of unpatentability. Paper 11 (“Dec.”). Those grounds are set forth in the following table:

<b>Claims challenged</b>	<b>Basis</b>	<b>Reference(s)<sup>1</sup></b>
10-20	§ 102	Szum
10-20	§ 103	Szum
1-9	§ 103	Szum and Shustack
1, 2, 8, 10, 11, 16, 17, and 19	§ 102	Shustack
1, 2, 8, 10, 11, 16, 17, and 19	§ 103	Shustack
3, 12, 18, and 20	§ 103	Shustack and Jackson

After institution of trial, DSM filed a patent owner response. Paper 43 (“PO Resp.”). DSM also filed a motion to amend claims by submitting proposed new claims 21 and 22 to be substituted for original claims 1 and 10, respectively. Paper 44 (“Mot. to Amend”). Corning filed a reply to the patent owner response (Paper 61 (“Reply”)) and also an opposition to DSM’s motion to amend (Paper 60). DSM then filed a reply in support of its motion to amend. Paper 72.

<sup>1</sup> The references are: WO 95/15928 (Ex. 1002) (“Szum”); U.S. Patent No. 5,352,712 (Ex. 1003) (“Shustack”); and U.S. Patent No. 4,900,126 (Ex. 1005) (“Jackson”).

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Both parties filed and fully briefed motions to exclude evidence. Paper 78 (“Pet. Mot. to Exclude”); Paper 75 (“PO Mot. to Exclude”). Oral hearing was held February 11, 2013. Paper 89 (“Transcript”).

The Board has jurisdiction under 35 U.S.C. § 6(c). This final written decision is issued pursuant to 35 U.S.C. § 318(a) and 37 C.F.R. § 42.73.

Corning fails to show by a preponderance of evidence that any of the challenged claims 1-20 are unpatentable. DSM’s motion to amend is denied, without prejudice. DSM’s motion to exclude evidence is denied. Corning’s motion to exclude evidence is dismissed as moot.

*B. Related Proceedings*

Corning and DSM are involved simultaneously in nine other *inter partes* reviews based on patents claiming similar subject matter: IPR2013-00043; IPR2013-00044; IPR2013-00046; IPR2013-00047; IPR2013-00048; IPR2013-00049; IPR2013-00050; IPR2013-00052; IPR2013-00053.

*C. The ’666 Patent (Ex. 1001)*

The ’666 patent generally relates to radiation-curable coating compositions for optical glass fibers commonly used in data transmission. Ex. 1001, 1:22-23. In particular, the patent describes optical glass fibers coated with two radiation-cured coatings. *Id.* at 1:30-31. An inner primary coating contacts the glass surface of the fiber. *Id.* at 1:32-34. An outer primary coating overlays the inner coating. *Id.* For identification purposes, the outer primary coating includes colorant or, alternatively, a third colored layer, called an ink coating, which is applied to the outer primary coating. *Id.* at 1:57-62.

Figure 1, depicting such an optical glass fiber, is reproduced below:

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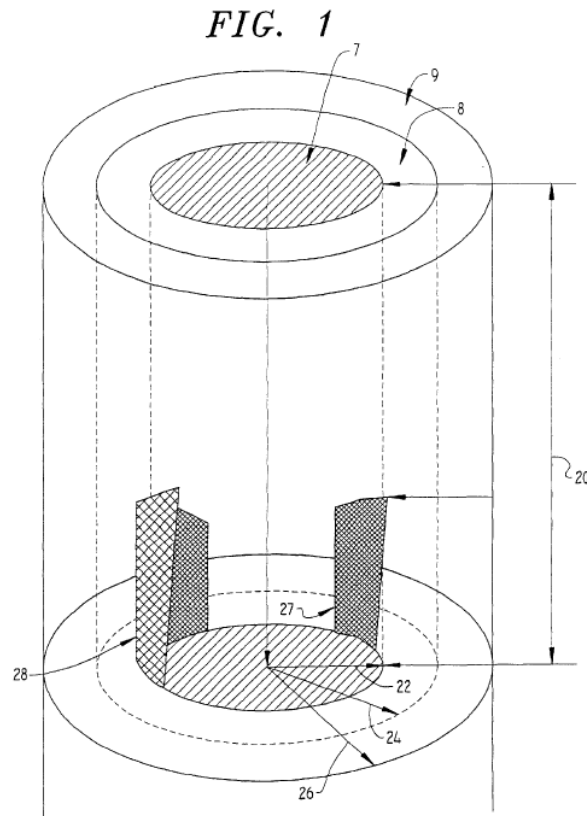


Figure 1 is a longitudinal cross-sectional view of coated optical glass fiber 7 coated with inner primary coating 8 and commercially available outer primary coating 9. *Id.* at 8:11-12; 10:10-12.

To create a cable or ribbon assembly, used in the construction of multi-channel transmission cables, a plurality of coated optical fibers are bonded together in a matrix material. *Id.* at 1:44-50. In order to connect the fibers of multiple ribbons, the surface of a glass fiber must be accessible. *Id.* at 1:57-2:10. That is often accomplished by a process known as “ribbon stripping”—removing the coatings and the matrix material, preferably as a cohesive unit. *Id.* The invention of the '666 patent is directed to a ribbon assembly having improved ribbon stripping capabilities. *Id.* at 1:25-27.

As described in the Background of the Invention, the prior art discloses ribbon assemblies composed of multiple optical glass fibers with

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both an inner and outer coating and an optional outer ink layer. *Id.* at 1:44-52. The two compositions used as the inner and outer coatings often are modified to provide desired properties—providing bare optical glass fibers, which, when stripped, are substantially free of residue. *Id.* at 2:37-4:33. For example, the inner primary coating may be modified to reduce adhesion between the coating and the optical glass fiber. *Id.* at 2:42-44. A reduction in adhesion facilitates easy removal of the coating during stripping, but also increases the possibility of undesirable delamination in the presence of moisture. *Id.* at 2:44-53. “Delamination of the inner primary coating from the optical glass fiber can lead to degraded strength of the optical glass fiber as well as signal transmission attenuation disadvantages.” *Id.* at 2:54-57.

We focus our analysis on a dispositive issue concerning a property, required by each challenged claim, of providing “sufficient adhesion to [a] glass fiber to prevent delamination in the presence of moisture and during handling.” *Id.* at claims 1, 10.

#### *D. Illustrative Claims*

Claims 1 and 10 are the only independent claims and are reproduced below (emphases added):

1. A composition for coating an optical fiber, said composition comprising propoxylated nonyl phenol acrylate and an oligomer having at least one functional group capable of polymerizing under the influence of radiation, said composition after radiation cure having the combination of properties of:
  - (a) a fiber pull-out friction of less than 40 g/mm at 90° C.;
  - (b) a crack propagation of greater than 1.0 mm at 90° C.;
  - (c) a glass transition temperature of below 10° C.; and
  - (d) *sufficient adhesion to said glass fiber to prevent delamination in the presence of moisture and during handling.*

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10. A composition for coating an optical fiber, said composition comprising an oligomer having an aliphatic diisocyanate residue and at least one functional group capable of polymerizing under the influence of radiation, said composition after radiation cure having the combination of properties of:

- (a) a fiber pull-out friction of less than 40 g/mm at 90° C.;
- (b) a crack propagation of greater than 1.0 mm at 90° C.;
- (c) a glass transition temperature of below -20° C. or less; and
- (d) *sufficient adhesion to said glass fiber to prevent delamination in the presence of moisture and during handling.*

## II. ANALYSIS

### A. Claim Construction

In an *inter partes* review, claim terms in an unexpired patent are interpreted according to their broadest reasonable construction in light of the specification of the patent in which they appear. 37 C.F.R. § 100(b); Office Patent Trial Practice Guide, 77 Fed. Reg. 48,756, 48,766 (Aug. 14, 2012). Claim terms are given their ordinary and customary meaning, as understood by one of ordinary skill in the art in the context of the entire disclosure. *In re Translogic Tech., Inc.*, 504 F.3d 1249, 1257 (Fed. Cir. 2007).

If an inventor acts as his or her own lexicographer, the definition must be set forth in the specification with reasonable clarity, deliberateness, and precision. *In re Paulsen*, 30 F.3d 1475, 1480 (Fed. Cir. 1994). The construction that stays true to the claim language and most naturally aligns with the inventor's description is likely the correct interpretation. *Renishaw PLC v. Marposs Società per Azioni*, 158 F.3d 1243, 1250 (Fed. Cir. 1998).

Each challenged claim requires a curable coating composition that, after radiation cure, exhibits “sufficient adhesion to [a] glass fiber to prevent delamination in the presence of moisture and during handling.” Ex. 1001,



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claims 1, 10. We refer to that property in our analysis as “the claimed adhesion property.”

The parties disagree about the meaning of the term “in the presence of moisture,” which appears in the limitation relating to the claimed adhesion property. Corning argues that the term is broad enough to embrace exposure to 95% relative humidity as disclosed in the ’666 patent for a wet adhesion test. Pet. 15; *see* Ex. 1001, 28:57–29:7 (disclosing conditions of wet adhesion test). DSM counters that “in the presence of moisture” means exposure to liquid water—that is, 100% relative humidity—as would be present, for example, in the water soak delamination test that is described in the ’666 patent. PO Resp. 24-25 (citing Ex. 2032 ¶¶ 46-53). That delamination test involves soaking a cured coating sample in a hot water bath for up to 24 hours. *See* Ex. 1001, 27:32-47; Table 3 (describing conditions of the water soak delamination test). DSM produces evidence that, under conditions of 95% relative humidity, “by definition, there will be no moisture condensation on the surface of the coating because moisture condenses at 100% relative humidity.” Ex. 2032 ¶ 48 (declaration of DSM’s witness, Dr. Carl R. Taylor).

The evidence supports a conclusion that the broadest reasonable interpretation of the term “moisture” is liquid water—that is, a condition of 100% relative humidity. The written description uses the term “moisture” in a context that suggests liquid water. *See, e.g.*, Ex. 1001, 29:5-7 (applying a “wax/water slurry” to surface of sample film in order “to retain moisture”); 35:24-26 (applying heat to remove “moisture” from samples, suggesting removal of liquid water). Moreover, where the written description discusses water in vapor form, the inventors use the word “humidity” or “atmospheric

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moisture,” but not “moisture” alone. *See, e.g., id.* at 21:54 (referring to “atmospheric moisture”); 28:55, 67; 29:5 (referring to “humidity”).

The ’666 patent further discloses that a “ribbon assembly can be buried under ground or water for long distance connections, such as between cities,” which is consistent with the proposition that an optical fiber coating must endure long periods of immersion in liquid water without delaminating. Ex. 1001, 67:20-22. In light of the context in which the term “moisture” appears in the specification, we conclude that the inventors used that term in its ordinary sense to refer to liquid water.

The ’666 patent, thus, is directed to a coating composition that, after radiation cure, has sufficient adhesion to glass to prevent delamination in the presence of liquid water. We decline to resolve what temperature, or length of time, of exposure to liquid water the coating must endure, without delaminating, in order to satisfy the claimed adhesion property. Resolving those conditions is not necessary to our analysis, which focuses on whether Corning’s wet adhesion test, conducted under conditions of 95% relative humidity, is probative of the extent to which a cured coating delaminates from glass when exposed to liquid water.

*B. Basis of Decision to Institute*

We instituted trial based on the limited information advanced in the petition and preliminary response. In the petition, Corning relied on wet adhesion test data to show that certain prior art coatings inherently exhibit the claimed adhesion property. Pet. 16, 18-19, 26. In the preliminary response, DSM did not challenge specifically Corning’s view that such data are sufficient to establish the claimed adhesion property in a cured coating. *See, e.g.,* Prelim. Resp. 8-12. Based on the record developed at the

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preliminary stage of the proceeding, therefore, the Board instituted trial. *See, e.g.*, Dec. 10 (citing Prelim. Resp. 8-12) (“DSM does not dispute, specifically, any of [Corning’s] assertions” regarding whether the properties required by the claims are inherently met in the prior art).

Additional information developed during the trial, however, casts doubt on the adequacy of wet adhesion test data to support a finding that the prior art coatings in fact exhibit the claimed adhesion property. Informed by the totality of evidence developed during the trial phase, and for the reasons set forth below, we conclude that Corning fails to show by a preponderance of evidence that Szum or Shustack inherently describes or suggests a coating composition that satisfies the claimed adhesion property.

*C. Szum: Anticipation and Obviousness of Claims 10-20*

Corning argues that claims 10-20 are anticipated by, or would have been obvious over, Szum. Corning admits that Szum does not disclose expressly a coating composition having the claimed adhesion property. The crux of Corning’s argument is that Szum inherently discloses a coating composition having that property, as evidenced by a wet adhesion test carried out under conditions of 95% relative humidity on a coating prepared according to Szum’s Example 5 (“the Szum coating”). Pet. 18-19.

DSM responds that the wet adhesion test does not evaluate for delamination, which is caused by exposure to liquid water, and that a different test—the water soak delamination test—is the only method disclosed in the ’666 patent for assessing delamination. *See* PO Resp. 16 (citing Ex. 2032 ¶¶ 46-53 (opinion of DSM’s witness, Dr. Taylor)). DSM also comes forward with its own test results, which allegedly show that the

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Szum coating, in fact, delaminates when subjected to the conditions of the water soak delamination test disclosed in the '666 patent. PO Resp. 29-30.

A dispositive question thus arises: Does Corning show by a preponderance of evidence that the Szum coating exhibits sufficient adhesion to prevent delamination from glass in the presence of liquid water? For the reasons set forth below, we answer that question in the negative. We first address the conditions set forth in the '666 patent for the wet adhesion test and the water soak delamination test. We then consider whether the wet adhesion test, which Corning performed on the Szum coating, is probative of the claimed adhesion property. Finally, we explain why an evaluation of DSM's water soak delamination test data is not necessary to our analysis.

*i. The Wet Adhesion Test*

The '666 patent describes a wet adhesion test for evaluating a cured coating sample on a glass substrate. Ex. 1001, 28:60-65. The wet adhesion test is conducted “at a temperature of  $23\pm 2^{\circ}$  C. and a relative humidity of  $50\pm 5\%$  for a time period of 7 days.” *Id.* at 28:66–29:1. A portion of the sample film is then “further conditioned at a temperature of  $23\pm 2^{\circ}$  C. and a relative humidity of 95% for a time period of 24 hours.” *Id.* at 29:2-5. During that step, “[a] layer of polyethylene wax/water slurry [is] applied to the surface of the further conditioned film to retain moisture.” *Id.* at 29:5-7.

The written description makes plain that the wet adhesion test assesses a cured coating that is conditioned at 95% relative humidity. *Id.* at 28:66–29:1. Corning acknowledges that fact. *See, e.g.*, Pet. 15 (citing Ex. 1006 ¶ 82) (“The term ‘wet adhesion’ is described in the '666 patent at col. 28, lines 53-58 as adhesion at 95% relative humidity.”). Corning raises no argument that application of a layer of “wax/water slurry” to the surface

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of the coating represents an exposure to 100% relative humidity.

Ex. 1001, 29:5-7; *see* Reply 3 (The wet adhesion test described in the '666 patent relates to conditioning “at 95% relative humidity—not liquid water immersion.” (citing Ex. 1001, 29:5-7)).

After conditioning the sample at 95% relative humidity, sample that appears “uniform and free of defects” is “peeled back from the glass.” Ex. 1001, 29:13-17. The wet adhesion test is performed on the peeled-back sample using a device that includes “a horizontal support and a pulley.” *Id.* at 29:8-12. With the glass secured to the horizontal support, a wire is “attached to the peeled-back end of the sample, run along the specimen and then run through the pulley in a direction perpendicular to the specimen.” *Id.* at 29:17-21. A wet adhesion value is determined by clamping the free end of the wire “in the upper jaw of the testing instrument,” which is activated “until the average force value, in grams force/inch,” becomes “relatively constant.” *Id.* at 29:21-24. The '666 patent discloses that “[t]he preferred value for wet adhesion is at least about 5 g/in.” *Id.* at 29:24-25.

On this record, we find that the wet adhesion test assesses the mechanical force required to peel a cured coating away from a glass substrate, after conditioning the coating at 95% relative humidity.

*ii. The Water Soak Delamination Test*

The '666 patent also discloses a water soak delamination test in which “coated microscope slides [are] soaked in [] water.” *Id.* at 27:32, 43. The samples are soaked in a beaker of deionized water that is placed in a 60° C. hot water bath. *Id.* at 27:43-45. The samples are “observed for delamination periodically. The time when the first signs of delamination” appear is recorded. *Id.* at 27:45-47.

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Table 2 in the '666 patent specification describes a “hot water soak” in which samples are “aged for 4 hours at 60° C.,” the water bath is “shut-off for about 70 hours,” and then the temperature is “brought back to 60° C. for an additional 48 hours.” *Id.* at 28:18-19, 23-25. The degree of delamination observed after the hot water soak is reported in Table 2 as “none” or “delam. [a]fter 1 hour at 60° C.” *Id.* at 28:15-29. Table 3 similarly reports results for a delamination test that is described as a “60° C. Water Soak.” *Id.* at 29:53. Delamination results are reported in terms such as “No Delamination After 24 Hours,” “Slight Delamination After 15 Minutes,” and “No Delamination After 8 Hours; Slight Delamination After 24 Hours.” *Id.* at 29:53-59.

On this record, we find that the water soak delamination test assesses the ability of a cured coating to withstand the hydrodynamic forces that cause delamination of a cured coating from a glass substrate in the presence of liquid water.

*iii. Corning Fails to Establish that the Szum Coating  
Inherently Exhibits the Claimed Adhesion Property*

The '666 patent discloses that the wet adhesion test evaluates the force required to peel a coating away from a glass substrate, after the coating has been conditioned at 95% relative humidity. Ex. 1001, 29:8-25.

The '666 patent identifies a different test—a water soak delamination test—for evaluating the extent of delamination that occurs when a cured coating is exposed to liquid water. *Id.* at 28:32-47. In DSM’s view, Corning fails to establish sufficiently that the wet adhesion test, or “[p]eel test,” can “be used to reliably determine what the results of a delamination test would be.”

PO Resp. 25. We agree.

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Corning prepared the Szum coating and subjected it to substantially the same wet adhesion test that is described in the written description of the '666 patent. *Compare* Ex. 1007 ¶¶ 41-44 (Declaration of Ms. Inna I. Kouzmina) (describing the wet adhesion test procedure performed on the Szum coating) *with* Ex. 1001, 28:57–29:25 (describing a wet adhesion test procedure performed on an inventive example). The '666 patent instructs, however, that coating samples are subjected to a water soak test and “examined for delamination” prior to conducting the wet adhesion test. Ex. 1001, 28:53-54. Specifically, the wet adhesion test is performed “[i]n addition” to the water soak delamination test. *Id.* at 28:52-56. It is the delamination test that ascertains “[t]he time when the first signs of delamination” appear in a coating sample that is immersed in water. *Id.* at 27:32-47.

Although the '666 patent describes a sequence of testing that includes both a delamination test and a wet adhesion test, Corning comes forward with no evidence that the Szum coating was subjected to a delamination test. *Id.*; *see* Ex. 1007 ¶¶ 41-43 (Corning’s test procedures). Corning’s own witness, Dr. Michael Winningham, was unaware of any delamination test performed by Corning on the Szum coating. PO Resp. 25 (citing Ex. 2029, 469:17–471:17). Corning relies on a wet adhesion value for the Szum coating that is expressed as a grams-per-inch mechanical force required to peel the coating away from a glass substrate after conditioning at 95% relative humidity. Ex. 1007 ¶¶ 41-44.

On this record, we find that Corning relies on test results obtained for the Szum coating after exposure to conditions of 95% relative humidity, but not to liquid water. As explained in our claim construction analysis,

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exposure to 95% relative humidity is not “in the presence of moisture” (i.e., liquid water) as specified in the challenged claims. Corning argues that the Szum coating exhibits a wet adhesion value “of 44 g/in when conditioned at 95% relative humidity,” but does not explain how that result is probative of adhesion in the presence of liquid water—that is, 100% relative humidity. Pet. 18 (citing Ex. 1006 ¶ 88); *see* Ex. 2032 ¶ 48 (moisture condenses at 100% relative humidity). On that basis, we determine that Corning fails to show by a preponderance of evidence that the Szum coating meets the claimed adhesion property.

A second independent basis supports our determination. Corning comes forward with evidence insufficient to support an inference that the results of a 95% relative humidity wet adhesion test correspond to an ability to withstand the hydrodynamic forces that effect delamination. Corning argues that Dr. Winningham “has confirmed that a coating composition with an adhesion to glass of either 23 g/in or 44 g/in, as in Szum, would have sufficient adhesion to the glass fiber to prevent delamination in the presence of moisture and during handling.” Pet. 18-19 (citing Ex. 1006 ¶ 88).<sup>2</sup> Dr. Winningham’s opinion on that point is unsupported and, therefore, unpersuasive. *See* Ex. 1006 ¶ 88 (reciting opinion without objective proof).

In that regard, Dr. Winningham repeats, verbatim, attorney argument set forth in the petition, but identifies no objective evidence explaining how a wet adhesion value, which indicates a mechanical force required to peel a coating away from a glass substrate, correlates to an ability to withstand the

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<sup>2</sup> In addition to the experimental test results tending to establish a wet adhesion value of 44 g/in for the Szum coating, Corning points to the reference itself for a teaching that the Szum coating exhibits “an adhesion to glass at 95% relative humidity of 23 g/in.” Pet. 18 (citing Ex. 1002, 25:12).



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hydrodynamic forces that effect delamination. *Id.* Dr. Winningham’s bare opinion is entitled to little weight in the absence of objective evidentiary support. *See Ashland Oil, Inc. v. Delta Resins & Refractories, Inc.*, 776 F.2d 281, 294 (Fed. Cir. 1985) (finding lack of factual support for expert opinion “may render the testimony of little probative value in a validity determination”).

During cross-examination, Dr. Winningham testified about the differences between a water soak delamination test and a wet adhesion test, which he refers to in his testimony as “a peel test”:

Q. If one was concerned about the ability of a coating to delaminate from a substrate when exposed to water, would performing a peel test not give sufficient information to satisfy the interested person?

A. I think those tests measure — are looking at different things or measuring different things, so I’m not sure if — I can’t say categorically that a peel test is going to tell you what’s going to happen in a water delamination test. Different tests.

Ex. 2029, 460:12-21.

Dr. Winningham also testified that the water soak delamination test evaluates the “hydrodynamic forces” that work to delaminate a coating from a glass substrate, whereas “a peel test” evaluates the “mechanical forces” exerted, where “one is applying a — mechanical force to a film and pulling the film off a substrate.” Ex. 2029, 459:3-18. That testimony of Corning’s witness is consistent with the explanation of the relevant hydrodynamic forces that is provided by DSM’s witness, Dr. Taylor. Ex. 2032 ¶¶ 52-53.

At the oral hearing, Corning’s counsel directed our attention to test results reported in Table 3 of the ’666 patent and, for the first time, argued that those results establish “a clear correlation between the films that passed

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the hot water soak test . . . and films that have a certain wet adhesion.”

Transcript 10:15-17. That argument, and Corning’s reliance on Table 3, is not set forth in the petition or the reply. *See* Pet. 18-19; Reply 2-5. We deem Corning to have waived that argument raised by counsel for the first time at the oral hearing. *Cf. Cross Med. Prods., Inc. v. Medtronic Sofamor Danek, Inc.*, 424 F.3d 1293, 1320-21 n.3 (Fed. Cir. 2005) (arguments not raised in an opening brief are deemed waived).

That argument also is unpersuasive because it is unsupported by convincing, objective evidence that explains a relationship between the wet adhesion values and the water soak delamination results reported in Table 3. In that regard, Corning asks us to infer a relationship between wet adhesion values (reported as a grams-per-inch mechanical force) and water soak delamination results (reported as an observation, for example, of slight delamination after 15 minutes) from Table 3. *See* Transcript 10:15-17; Ex. 1001, 29:50-59 (Table 3). That is a leap we will not undertake in the absence of persuasive evidence, such as a technical article or expert testimony, explaining some relationship between those disparate test results. Pet. 18-19; Reply 2-5 (identifying no such evidence).

Because the issue is not discussed in the briefs, moreover, we have no evidence as to how the wet adhesion value obtained for the Szum coating, which never endured a hot water soak, is comparable to the wet adhesion values reported in Table 3, which appear to relate to coating samples that endured both a hot water soak and the wet adhesion test. *See* Ex. 1001, 28:53-57 (wet adhesion test is performed “[i]n addition” to water soak delamination test); *see also id.* at 29:13-15 (after conditioning, “samples that appeared to be uniform and free of defects” were selected for the wet

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adhesion test, implying that samples that delaminated were excluded from such testing).

In sum, two independent reasons support our determination that the wet adhesion test results advanced by Corning fail to show adequately that the Szum coating has “sufficient adhesion . . . to prevent delamination in the presence of moisture” within the meaning of the challenged claims. First, the wet adhesion test assesses a property of the coating after conditioning at 95% relative humidity, which is not “in the presence of moisture.” Second, Corning identifies no persuasive evidence from which we reasonably can discern that the wet adhesion test evaluates for “delamination.”

*iv. DSM’s Delamination Test Results  
are Not Necessary to our Analysis*

DSM presents evidence that the Szum coating exhibits insufficient adhesion to prevent delamination in the presence of liquid water. Specifically, DSM contends that it formulated a coating according to Szum’s Example 5 and subjected it to the water soak delamination test described in the ’666 patent. PO Resp. 29-30 (citing Ex. 2032 ¶¶ 66-69). DSM reports that the Szum coating “experienced delaminations, visible to the unaided eye,” and that those “delaminations appeared as water-filled voids or ‘blisters’ between the inner primary coating and the glass.” *Id.* Corning counters that those results are unreliable because DSM failed to follow the correct procedure for preparing the Szum coating. Reply 5. In particular, Corning argues that DSM used the wrong photoinitiator in its formulation, which negatively affected the ability of the Szum coating to withstand delamination. *Id.* (citing Ex. 1063 ¶¶ 95-105).

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Corning bears the burden of showing by a preponderance of evidence that the Szum coating inherently discloses the claimed adhesion property. We need not resolve whether DSM properly formulated the Szum coating or whether DSM's test results accurately reflect the ability of that coating to withstand delamination in the presence of moisture. Our decision rests on Corning's failure to show sufficiently that its wet adhesion test results, which relate to the mechanical force sufficient to peel a coating away from a glass substrate after conditioning at 95% relative humidity, are probative of whether the Szum coating has "sufficient adhesion . . . to prevent delamination in the presence of moisture" (i.e., liquid water).

Based on the record developed at trial, Corning fails to show by a preponderance of evidence that the Szum coating inherently discloses the claimed adhesion property. Because each of claims 10-20 includes a limitation directed to that property, each claim withstands Corning's challenge based on anticipation by, or obviousness over, the Szum coating.

*D. Grounds of Unpatentability Based on Shustack*

We next turn to the other grounds of unpatentability raised at trial: (1) claims 1-9 based on obviousness over Szum and Shustack; (2) claims 1, 2, 8, 10, 11, 16, 17, and 19 based on anticipation by Shustack; (3) claims 1, 2, 8, 10, 11, 16, 17, and 19 based on obviousness over Shustack; and (4) claims 3, 12, 18, and 20 based on obviousness over Shustack and Jackson. *See* Dec. 22.

The first of those grounds is based on Corning's view that the wet adhesion test results establish that the Szum coating inherently exhibits the claimed adhesion property. Pet. 30-31, 33. For the reasons discussed above, Corning fails to establish that fact by a preponderance of evidence.

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The other grounds are based on a substantially similar argument that a coating made according to Example 1 of Shustack (“the Shustack coating”) exhibits the claimed adhesion property. Corning argues that it prepared the Shustack coating, subjected it to the 95% relative humidity wet adhesion test disclosed in the ’666 patent, and determined that it exhibits “adhesion to glass at 95% relative humidity of 77 g/in.” Pet. 39 (citing Ex. 1007 ¶¶ 30, 35, 40, 44; Ex. 1006 ¶ 121); *see* Ex. 1007 ¶¶ 41-44.

For reasons set forth above, the wet adhesion test results do not show sufficiently whether, or to what extent, a cured coating delaminates from glass in the presence of liquid water. On this record, Corning fails to show by a preponderance of evidence that the Shustack coating inherently discloses the claimed adhesion property. Because each challenged claim includes a limitation directed to that property, we are not persuaded that Corning carries its burden of demonstrating the unpatentability of any claim based on any ground advanced at trial.

*E. DSM’s Motion to Amend*

“A motion to amend may cancel a challenged claim or propose a reasonable number of substitute claims.” 37 C.F.R. § 42.121(a)(3). DSM moves to substitute claim 21 for original claim 1 and substitute claim 22 for original claim 10. Mot. to Amend 1-2. The overall framework of our amendment process is geared towards deciding motions to amend where the claims sought to be replaced are under a continuing threat of cancellation by virtue of the patentability challenge upon which trial is instituted. *See* 35 U.S.C. § 316(d) (tethering amendments to “challenged” patent claims and contemplating amendments that “materially advance” settlement of an ongoing dispute); *see also* 37 C.F.R. § 42.121(a)(2)(i) (an amendment may

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be denied where it “does not respond to a ground of unpatentability involved in the trial”). Absent some showing of special circumstances, we are not convinced that our charter of deciding cases quickly and efficiently favors dedicating our limited resources to evaluating the patentability of DSM’s proposed substitute claims, after we have resolved the main controversy in DSM’s favor.

Having determined that none of DSM’s original claims are proven unpatentable, we deny without prejudice the motion to amend. *See* Office Patent Trial Practice Guide, 77 Fed. Reg. at 48,767 (indicating that a motion to amend may be denied, without prejudice, if it is determined that patent owner’s original claims are not unpatentable). At the hearing, counsel stated that DSM is “conceding [c]laims 1 and 10.” Transcript, 19:4-10. To the extent counsel’s statement represents a request to cancel claims 1 and 10, we observe that a patent owner may disclaim a patent claim at any time during the life of a patent. 35 U.S.C. § 253; 37 C.F.R. § 1.321. Alternatively, a patentee may seek reissue if it believes that an error has occurred. 35 U.S.C. § 251. Our decision denying the motion to amend is without prejudice to the filing of a disclaimer or, if appropriate, seeking reissue.

*F. Motions to Exclude Evidence*

DSM moves to exclude Dr. Winningham’s testimony as unreliable, based on argument that he failed to review data underlying his opinions and, further, failed to apply the correct legal standard for obviousness. PO Mot. to Exclude 1-6. Because DSM’s argument goes to the weight that should be accorded the testimony, and not its admissibility, we decline to exclude that evidence. DSM also moves to exclude certain test results and testimony relating to fiber pull-out and dL/L testing, as well as certain exhibits

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submitted with Corning's reply. *Id.* at 7-14. We rely on none of that evidence to reach our final decision. We decline to exclude evidence that does not bear upon our decision.

On this record, DSM's motion to exclude evidence is denied.

Corning also moves to exclude evidence. Pet. Mot. to Exclude 1-11. Specifically, Corning seeks to exclude testimony relating to the water soak delamination tests that DSM performed on prior art coatings, as well as opinions relating to DSM's fiber pull-out friction tests. *Id.* We rely on none of that evidence to reach our final decision. Here again, we decline to exclude evidence that does not underlie our decision.

On this record, Corning's motion to exclude evidence is dismissed as moot.

### III. CONCLUSION

Corning has not shown by a preponderance of evidence that the subject matter of claims 10-20 of the '666 patent is anticipated by, or would have been obvious over, the teachings of Szum.

Corning has not shown by a preponderance of evidence that the subject matter of claims 1-9 would have been obvious over the combined teachings of Szum and Shustack.

Corning has not shown by a preponderance of evidence that the subject matter of claims 1, 2, 8, 10, 11, 16, 17, and 19 of the '666 patent is anticipated by, or would have been obvious over, the teachings of Shustack.

Corning has not shown by a preponderance of evidence that the subject matter of claims 3, 12, 18, and 20 would have been obvious over the combined teachings of Shustack and Jackson.

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DSM's motion to amend claims is *denied*.

The parties' motions to exclude evidence are *denied*.

This is a final decision. Parties to the proceeding seeking judicial review of the decision must comply with the notice and service requirements of 37 C.F.R. § 90.2.

#### IV. ORDER

It is

ORDERED that Corning's request to cancel claims 1-20 is *denied*;

FURTHER ORDERED that DSM's motion to amend claims is *denied*, without prejudice;

FURTHER ORDERED that DSM's motion to exclude evidence is *denied*; and

FURTHER ORDERED that Corning's motion to exclude evidence is *dismissed as moot*.



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(21) Appl. No.: **09/838,140**  
(22) Filed: **Apr. 20, 2001**

### Related U.S. Application Data

(63) Continuation of application No. 09/035,771, filed on Mar. 6, 1998, which is a continuation-in-part of application No. 08/877,585, filed on Jun. 17, 1997, now abandoned, which is a continuation-in-part of application No. 08/840,893, filed on Apr. 17, 1997, now abandoned, which is a continuation-in-part of application No. 08/745,790, filed on Nov. 8, 1996, now abandoned.

(51) **Int. Cl.**<sup>7</sup> ..... **G02B 6/22**; C08F 2/48

(52) **U.S. Cl.** ..... **385/128**; 385/141; 385/145;  
522/178

(58) **Field of Search** ..... 385/123–128,  
385/141, 143, 145; 522/172–183, 904, 905

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Properties of the R1055 coating composition, which was commercially available before November 1995.

Properties of the 950-106 coating composition, which was commercially available before November 1995.

Properties of the 3471-1-129 coating composition, which was commercially available before November 1995.

(List continued on next page.)

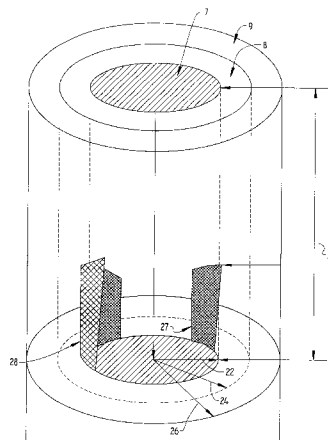
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(57) **ABSTRACT**

Optical fiber coatings are disclosed having excellent ribbon stripping and adhesion behavior. The coatings are radiation-curable. The excellent stripping and adhesion behavior can be achieved by several means which include by use of additives, by use of radiation-curable oligomers having higher molecular weight, or by use of coatings having certain thermal properties. Combination of means can be employed. Stripping behavior can be measured by crack propagation and fiber friction measurements.

**20 Claims, 11 Drawing Sheets**



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FIG. 1

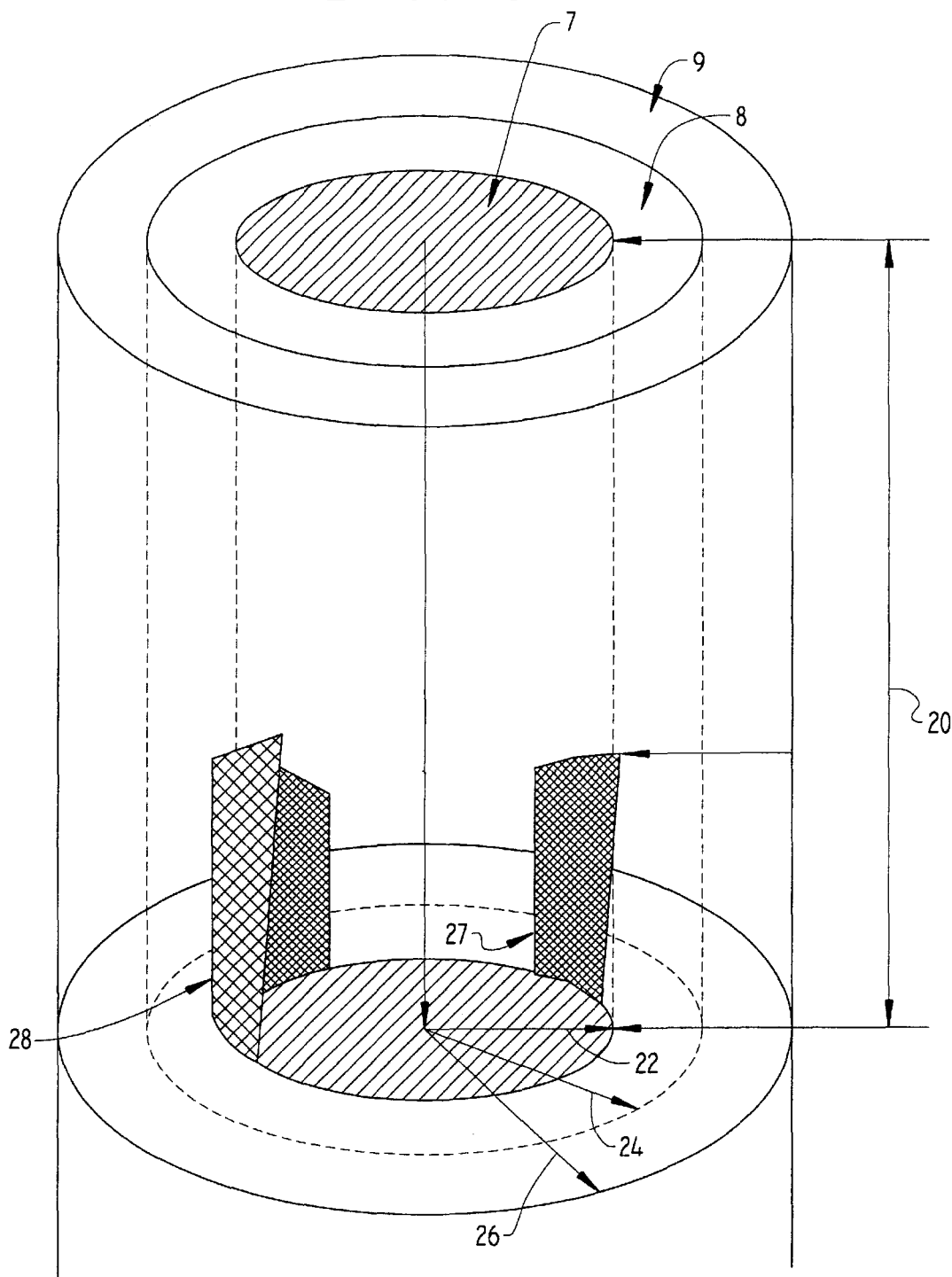
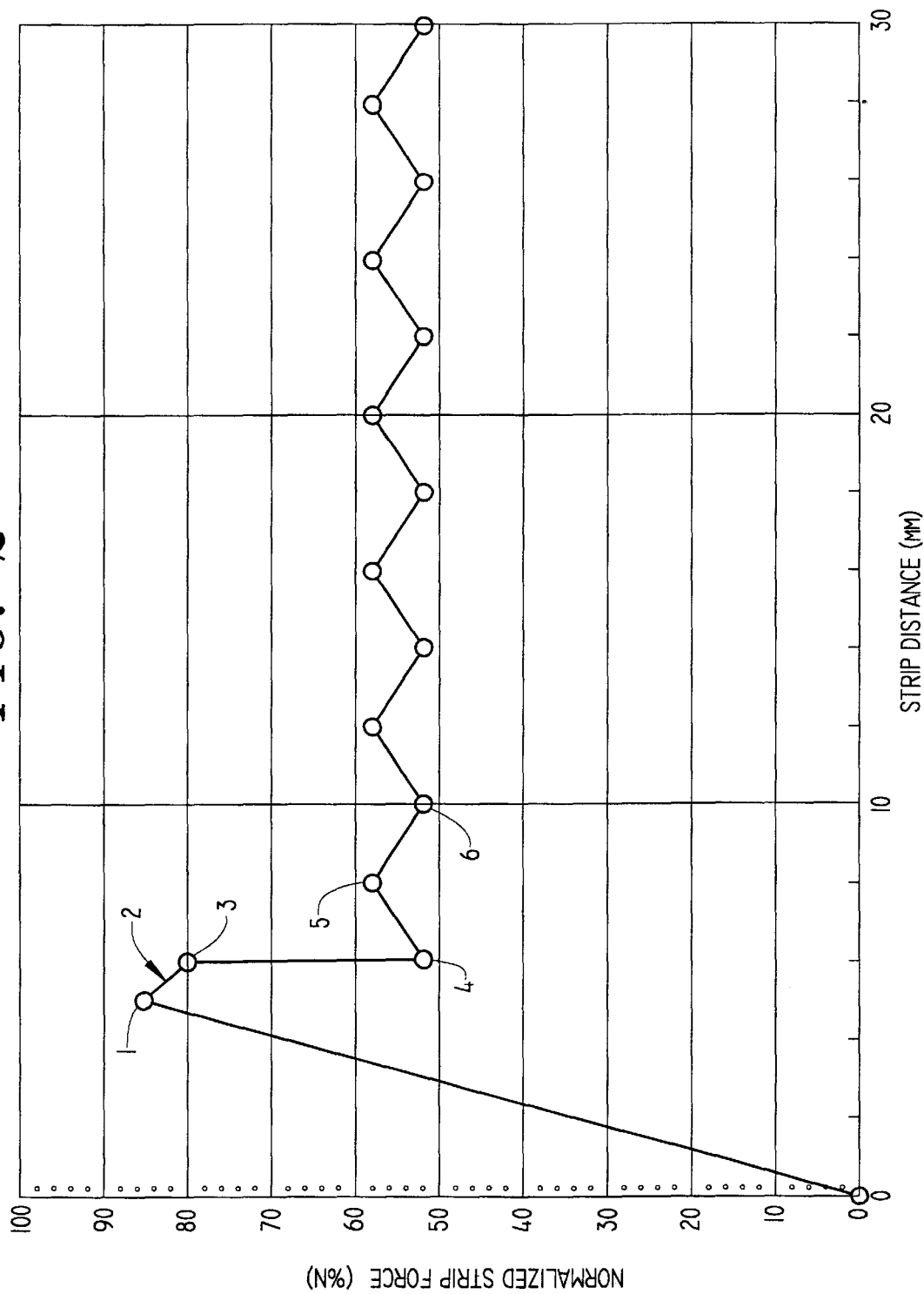


FIG. 2



**FIG. 3**

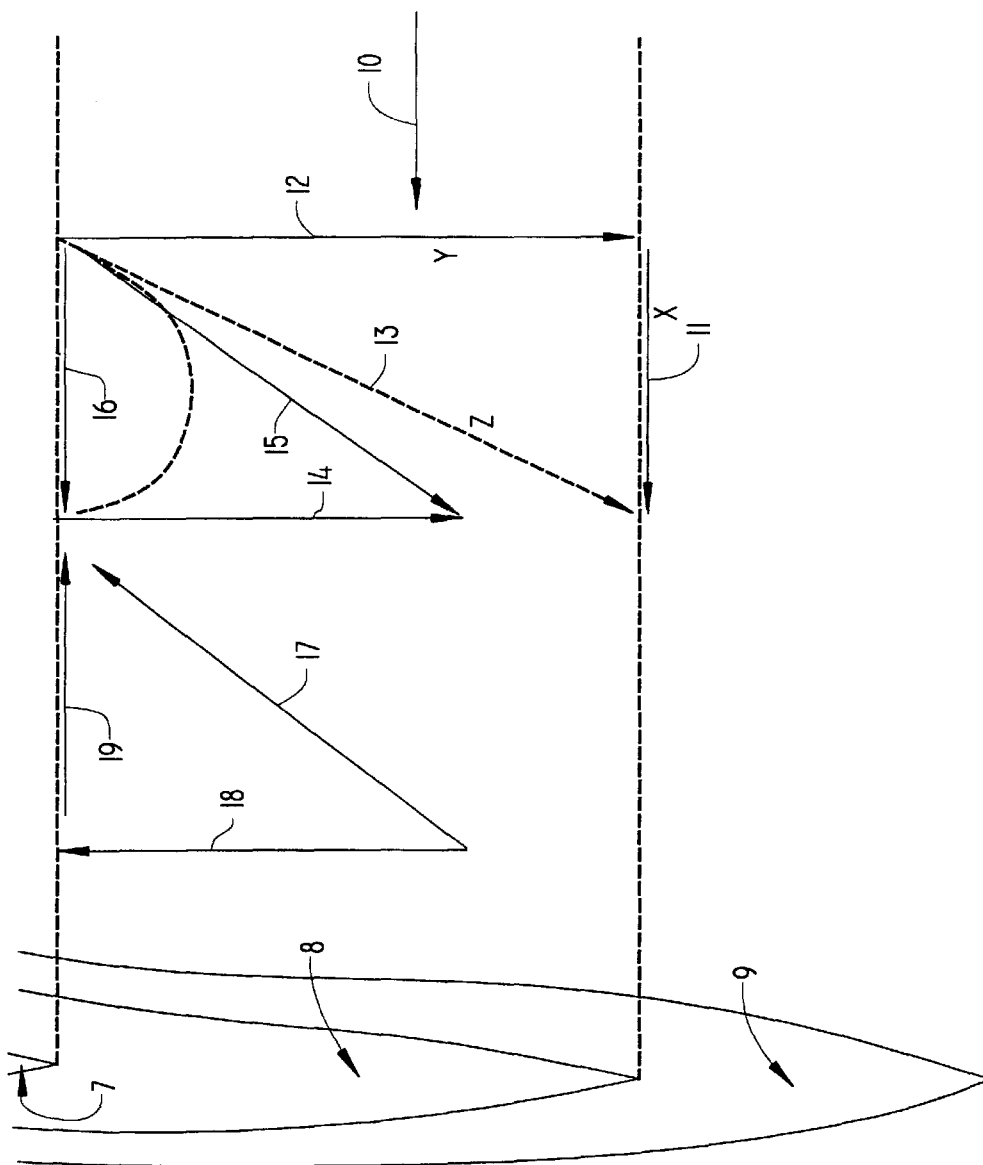


FIG. 4

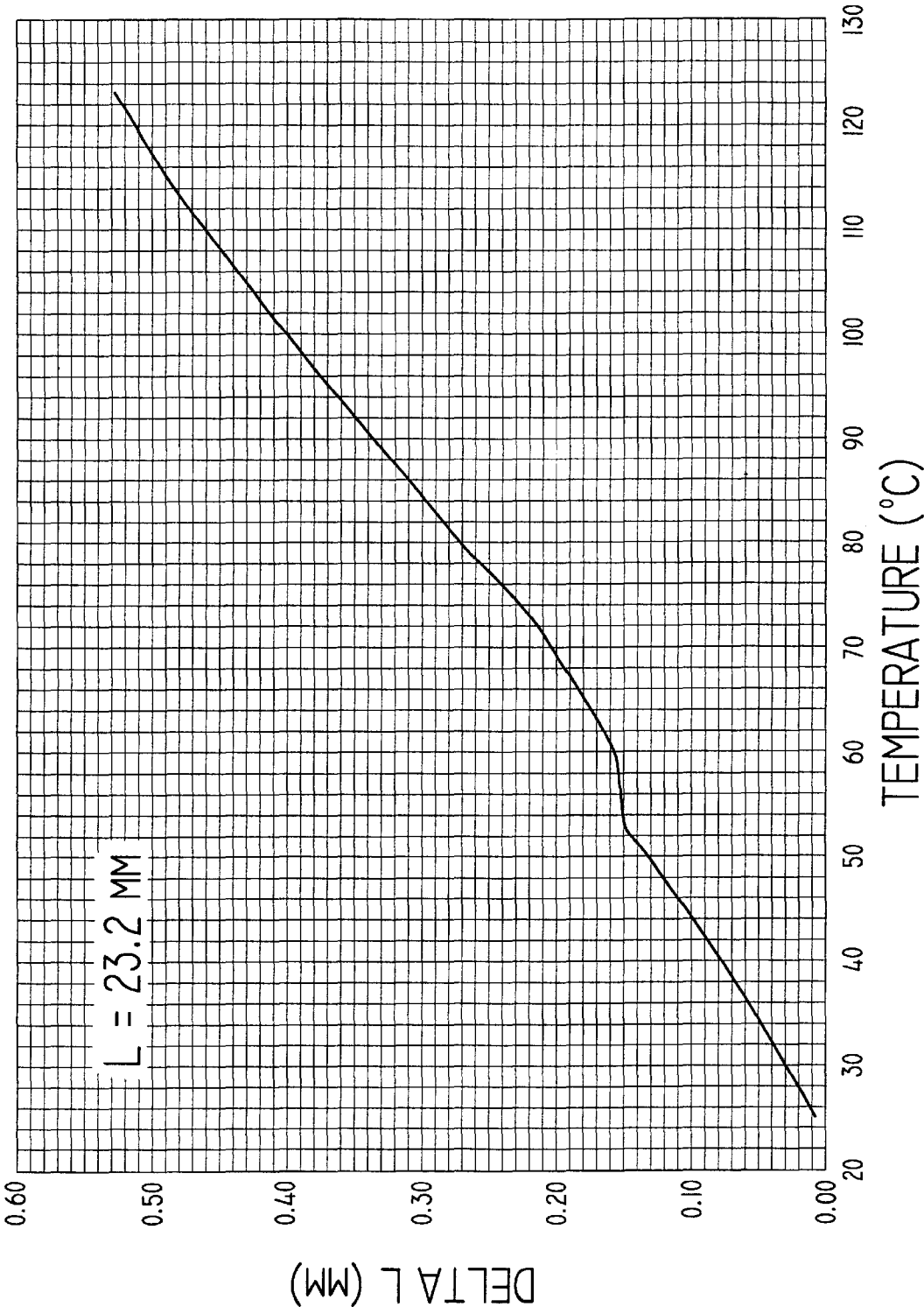


FIG. 5

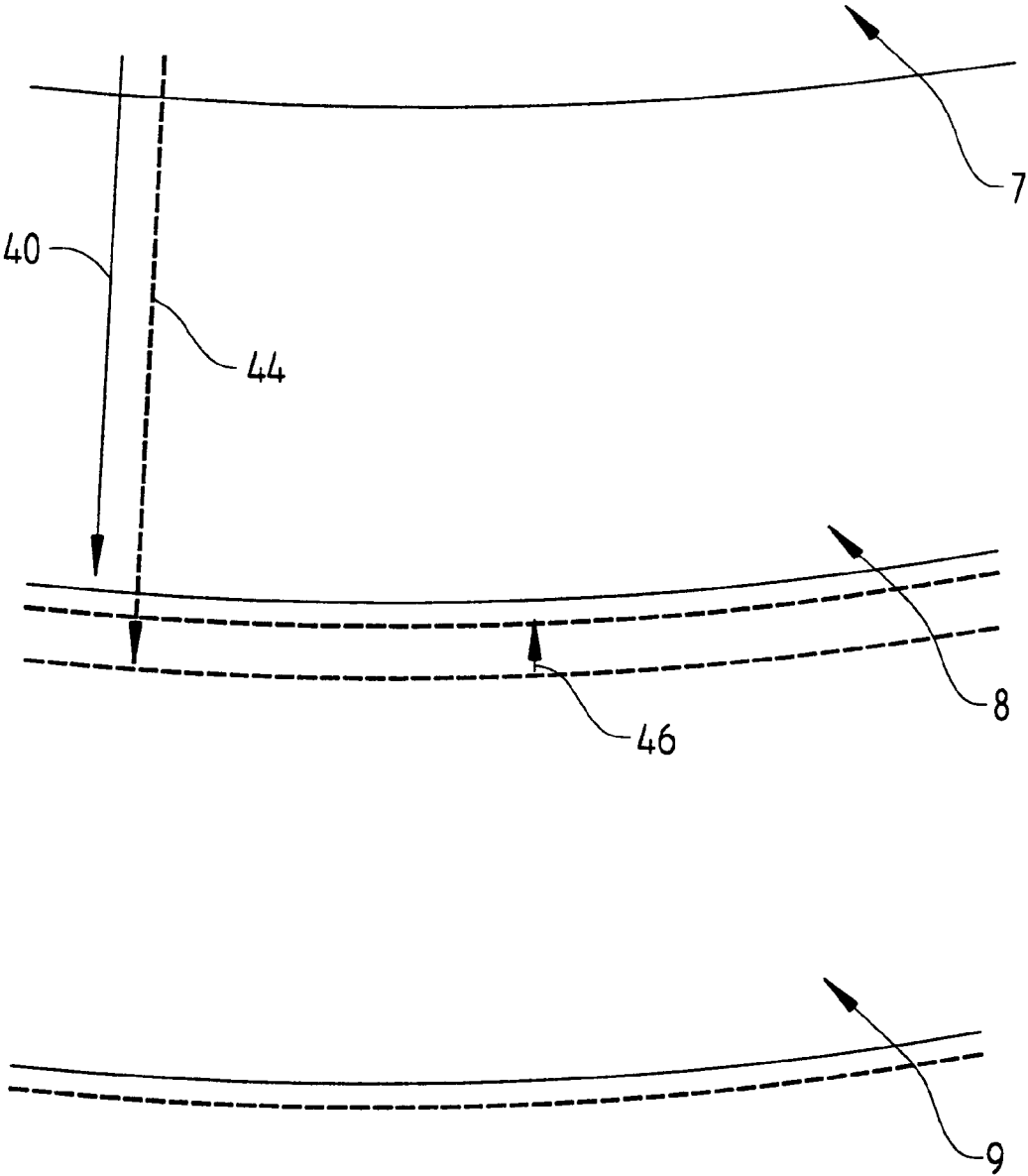




FIG. 6

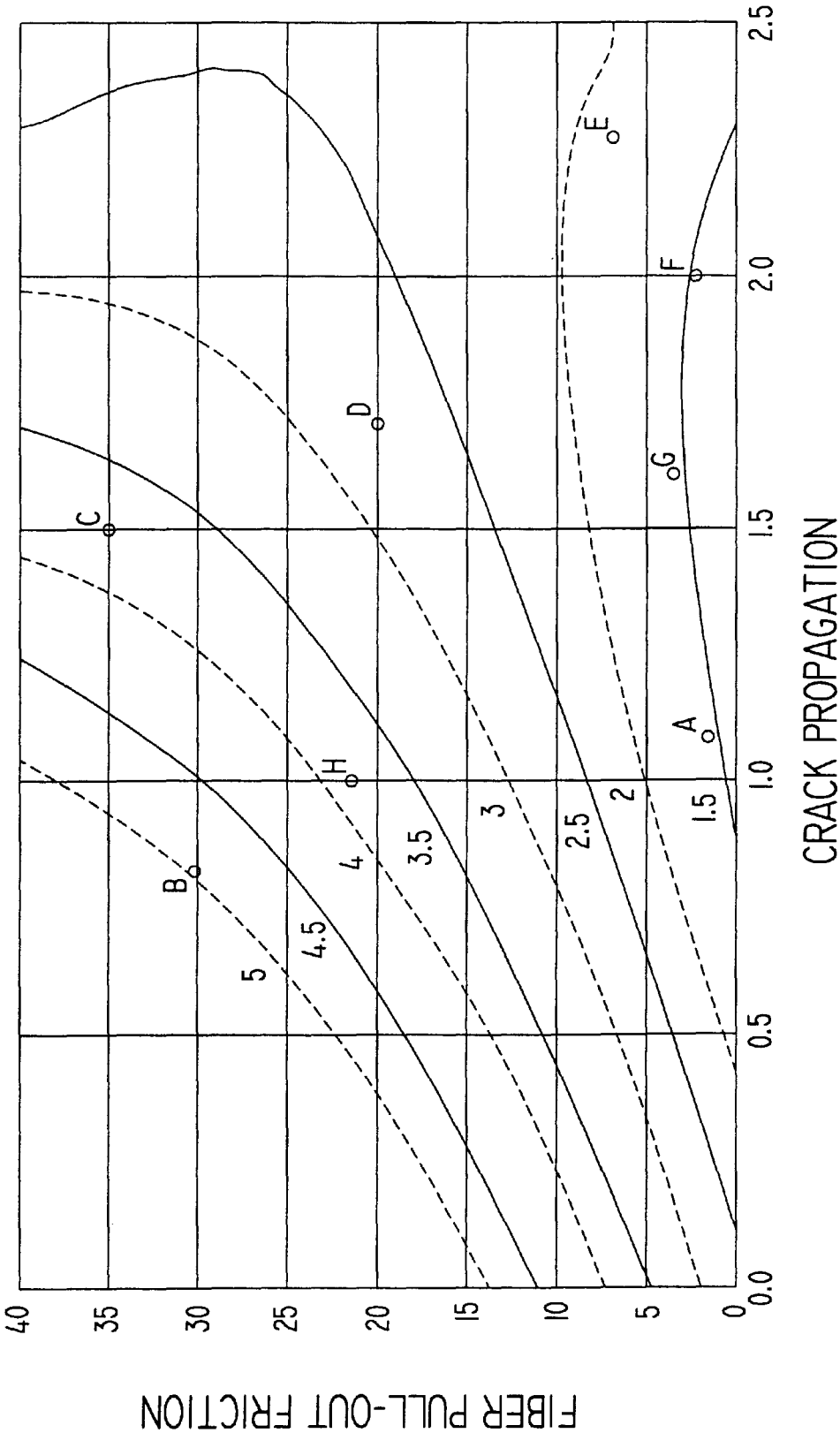


FIG. 7

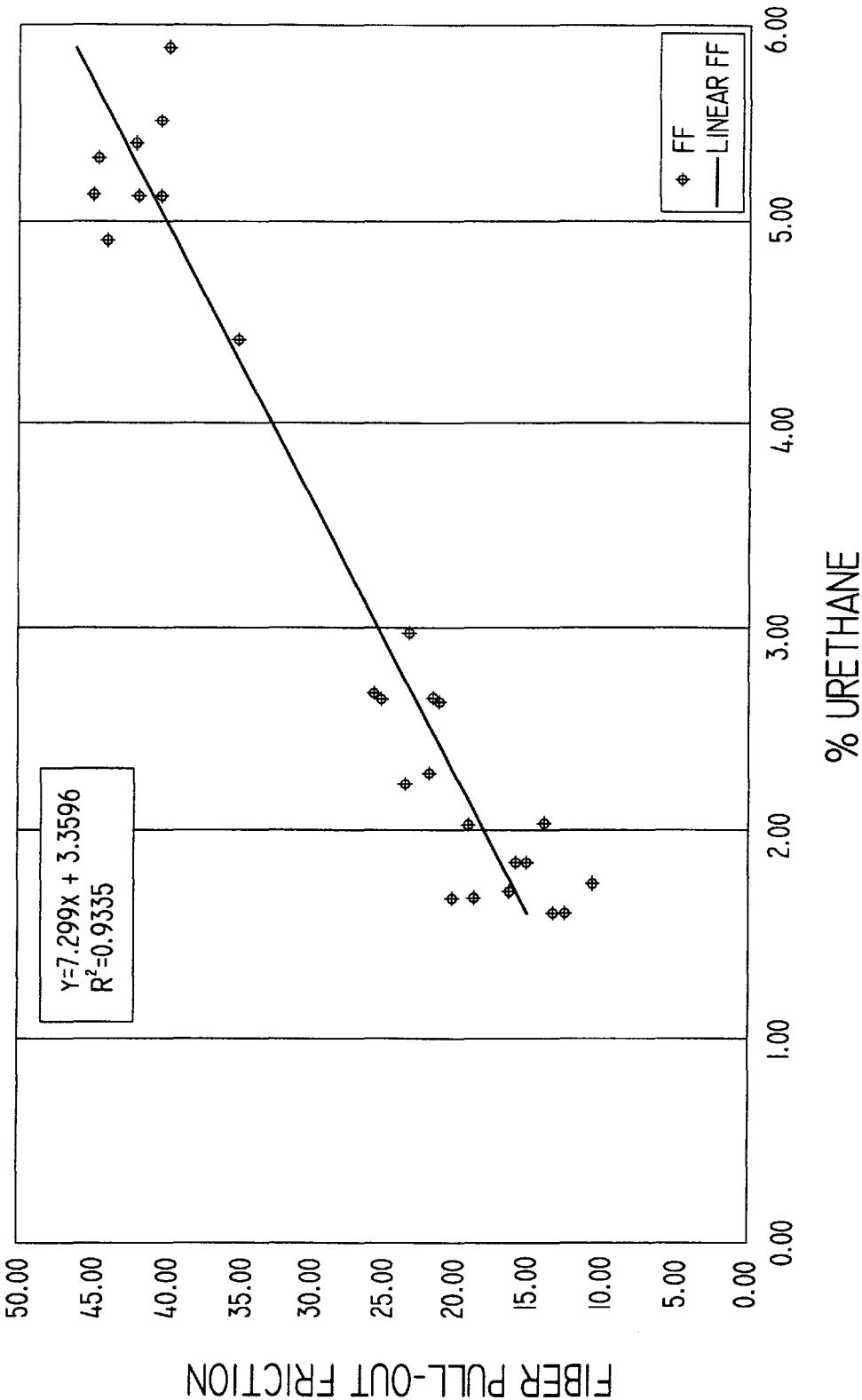


FIG. 8

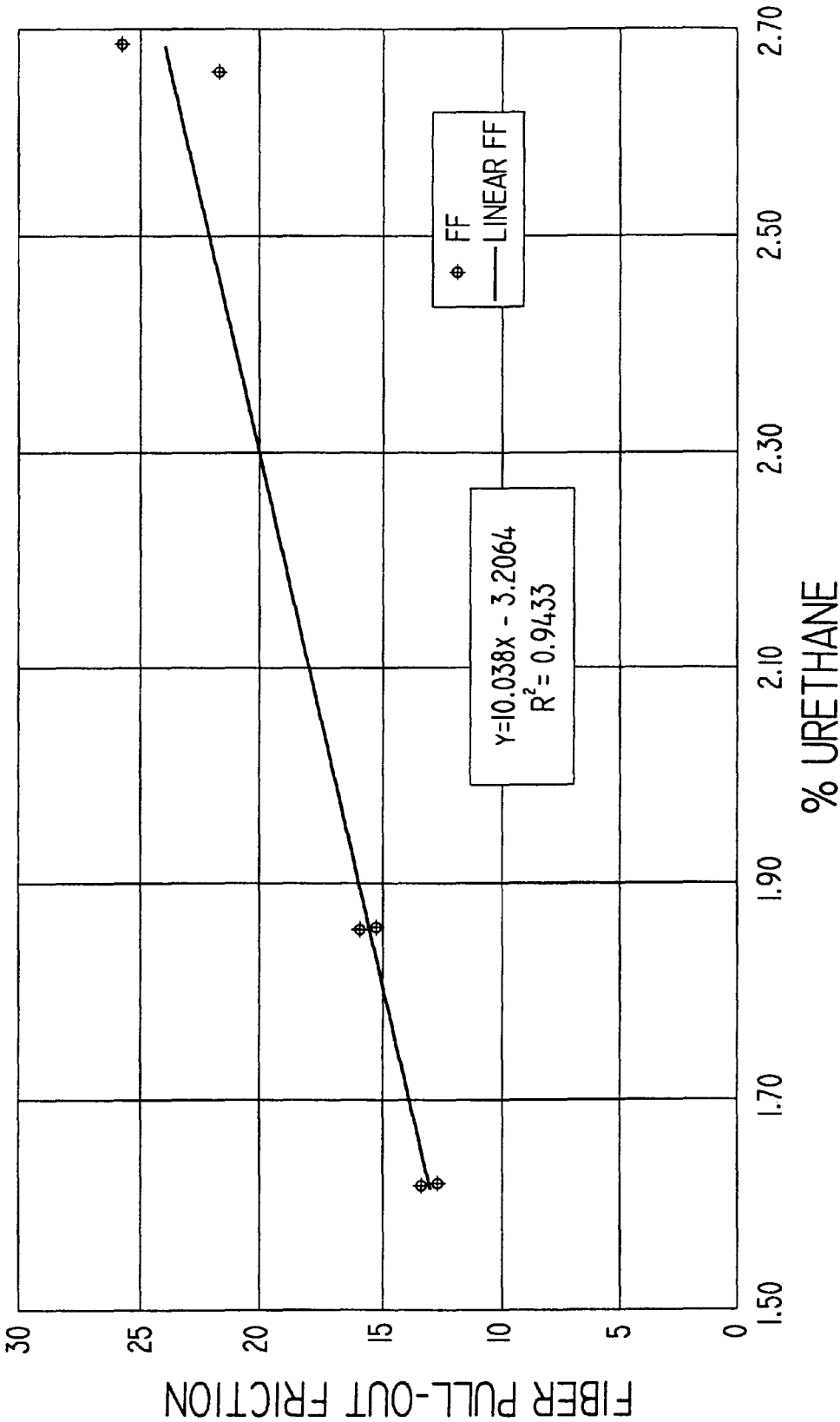


FIG. 9

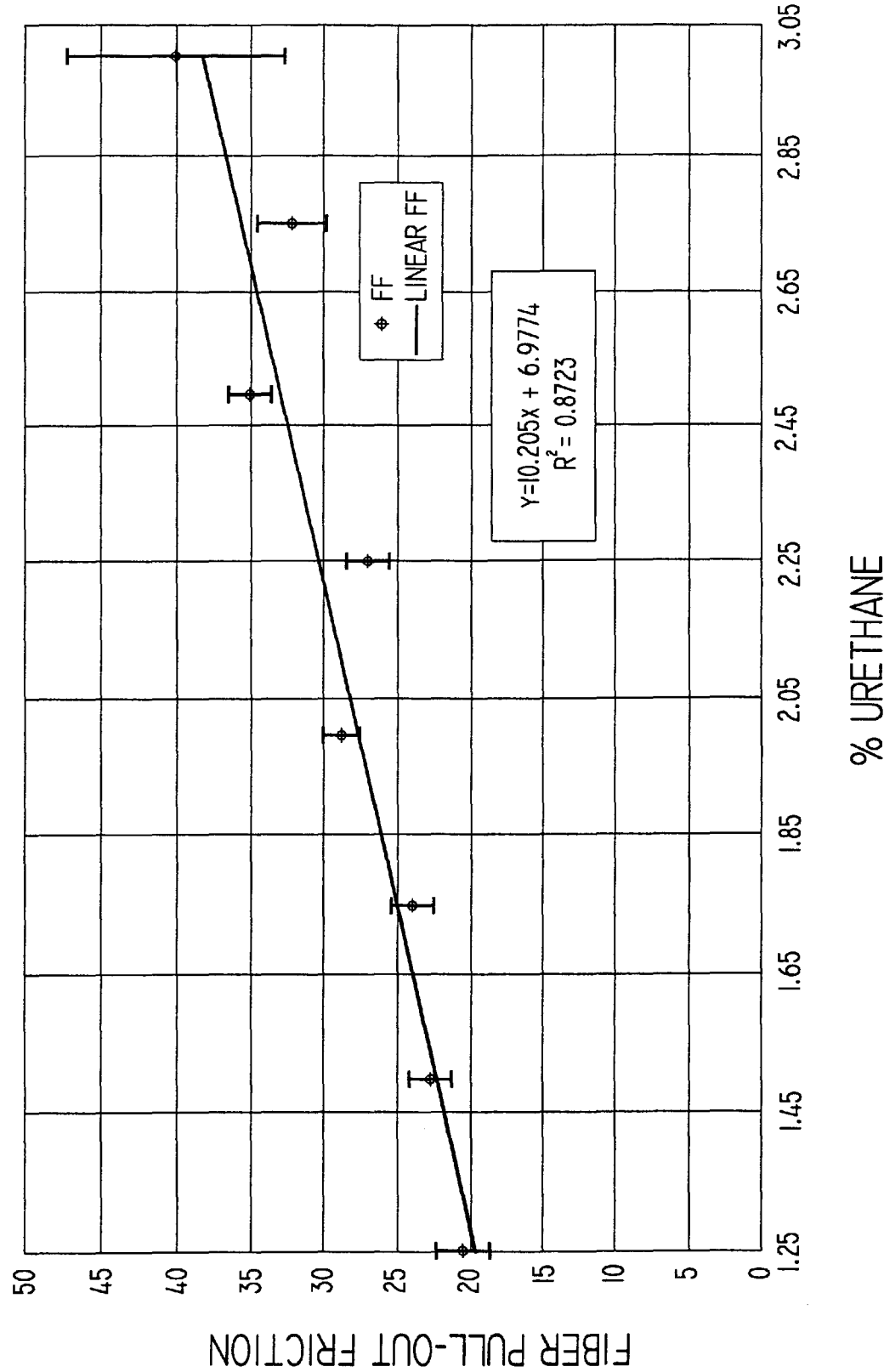


FIG. 10

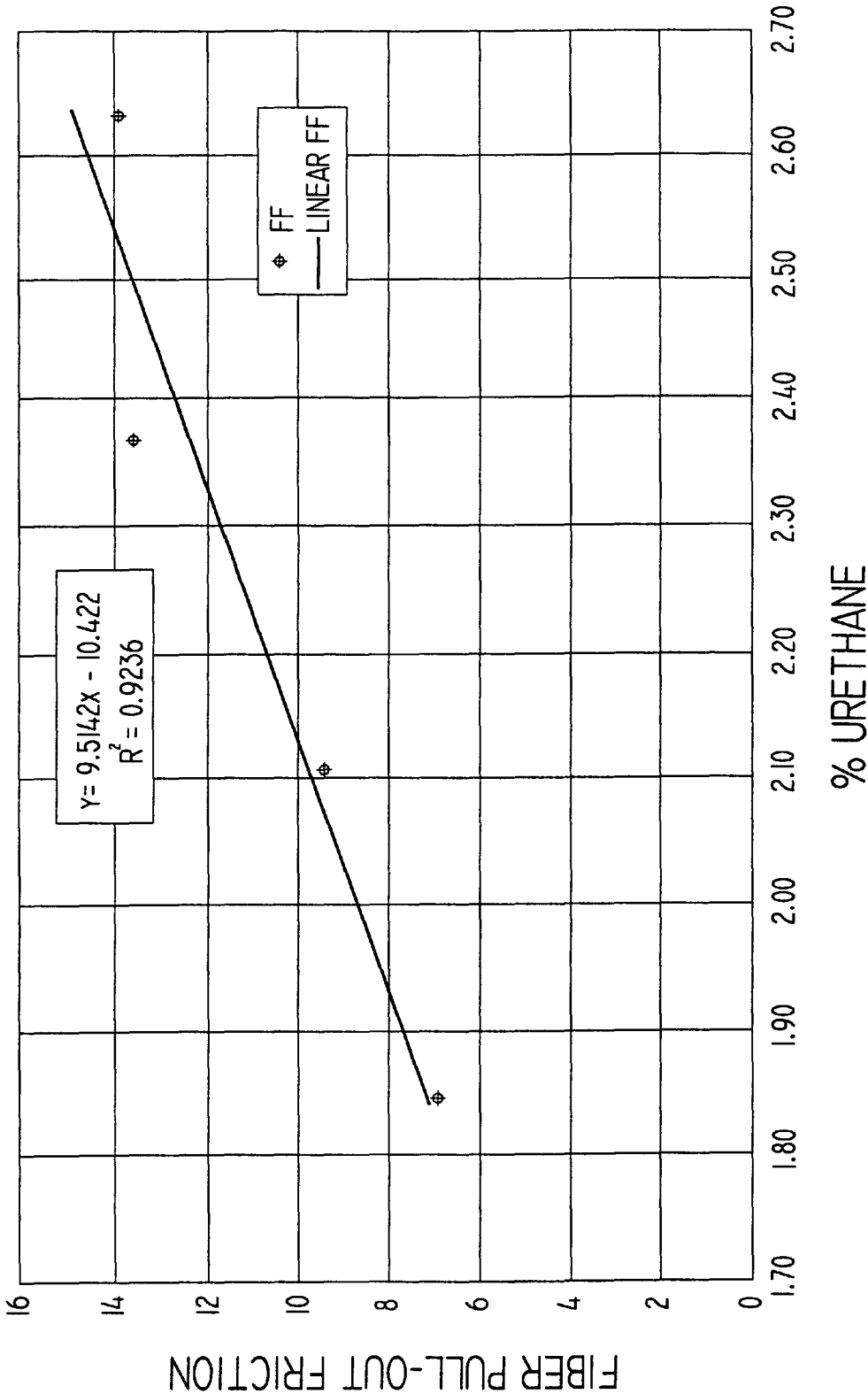
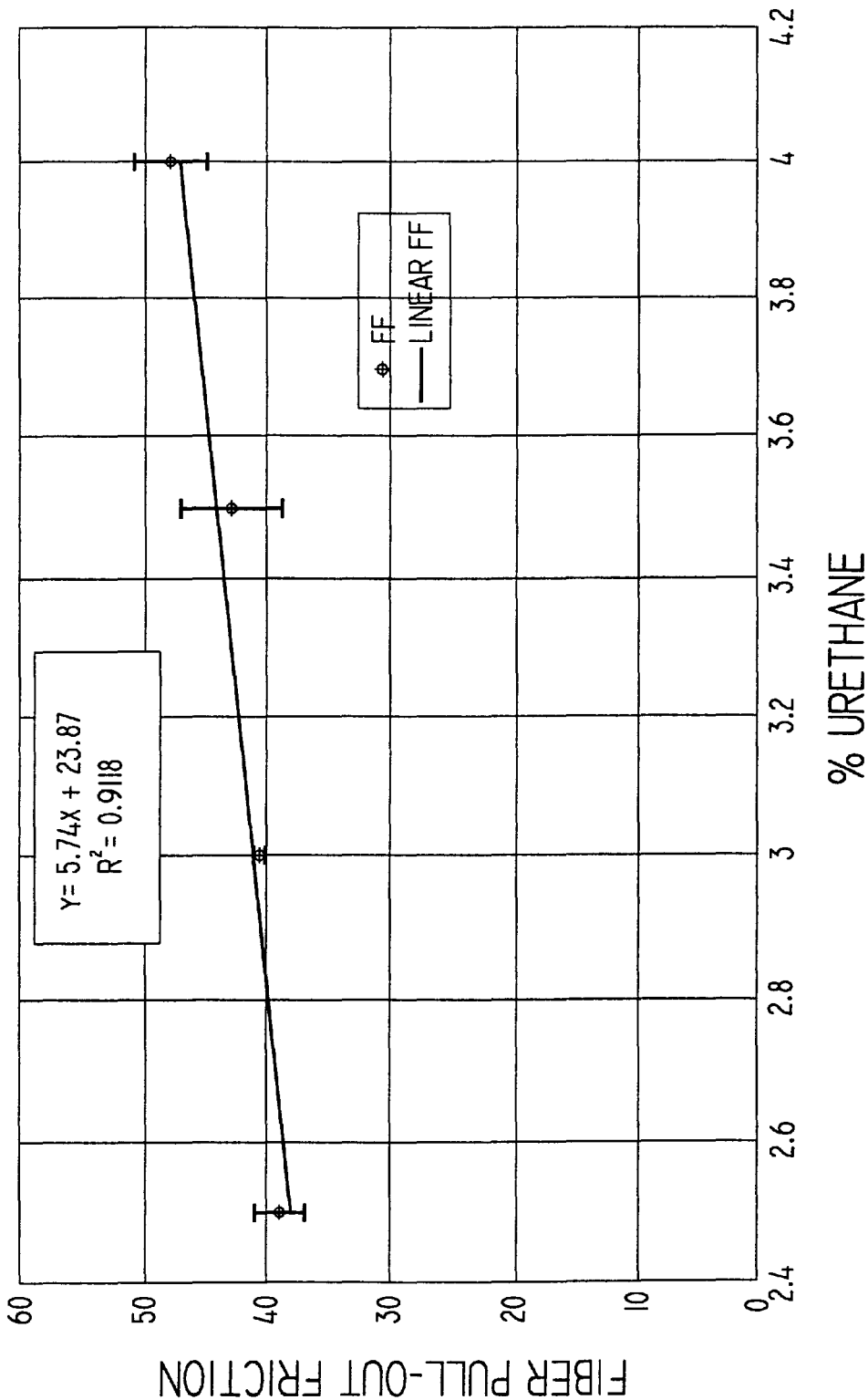


FIG. 11



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# **RADIATION-CURABLE OPTICAL GLASS FIBER COATING COMPOSITIONS, COATED OPTICAL GLASS FIBERS, AND OPTICAL GLASS FIBER ASSEMBLIES**

## **CROSS-REFERENCE TO RELATED APPLICATIONS**

This application is a continuation application of U.S. patent application Ser. No. 09/035,771, filed on Mar. 6, 1998, which is itself a continuation-in-part application of U.S. patent application Ser. No. 08/877,585, filed on Jun. 17, 1997 (abandoned), which is itself a continuation-in-part application of U.S. patent application Ser. No. 08/840,893 (abandoned), filed on Apr. 17, 1997, which is itself a continuation-in-part application of U.S. patent application Ser. No. 08/745,790 (abandoned), filed on Nov. 8, 1996, all of which are hereby incorporated in their entirety by reference

## **FIELD OF THE INVENTION**

The invention relates to radiation-curable inner and outer primary optical glass fiber coating compositions. The invention also relates to coated optical glass fibers and optical glass fiber assemblies. More particularly, the invention relates to a ribbon assembly having improved ribbon stripping capabilities.

## **BACKGROUND OF THE INVENTION**

Optical glass fibers are usually coated with two superposed radiation-cured coatings, which together form a primary coating. The coating which contacts the glass surface is called the inner primary coating and the overlying coating is called the outer primary coating.

The inner primary coating is usually a soft coating having a low glass transition temperature (hereinafter "T<sub>g</sub>"), to provide resistance to microbending. Microbending can lead to attenuation of the signal transmission capability of the coated optical glass fiber and is therefore undesirable. The outer primary coating is typically a harder coating providing desired resistance to handling forces, such as those encountered when the coated fiber is cabled.

For the purpose of multi-channel transmission, optical glass fiber assemblies containing a plurality of coated optical fibers have been used. Examples of optical glass fiber assemblies include ribbon assemblies and cables. A typical optical glass fiber assembly is made of a plurality of coated optical glass fibers which are bonded together in a matrix material. For example, the matrix material can encase the optical glass fibers, or the matrix material can edge-bond the optical glass fibers together.

Optical glass fiber assemblies provide a modular design which simplifies the construction, installation and maintenance of optical glass fibers by eliminating the need to handle individual optical glass fibers.

Coated optical glass fibers for use in optical glass fiber assemblies are usually coated with an outer colored layer, called an ink coating, or alternatively a colorant is added to the outer primary coating to facilitate identification of the individual coated optical glass fibers. Such ink coatings and colored outer primary coatings are well known in the art. Thus, the matrix material which binds the coated optical glass fibers together contacts the outer ink layer if present, or the colored outer primary coating.

When a single optical glass fiber of the assembly is to be fusion connected with another optical glass fiber, or with a

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connector, an end part of the matrix layer can be removed to separate each of the optical glass fibers.

Desirably, the primary coatings on the coated optical glass fibers, and the ink coating if present, are removed simultaneously with the matrix material to provide bare portions on the surface of the optical glass fibers (hereinafter referred to as "ribbon stripping"). In ribbon stripping, the matrix material, primary coatings, and ink coating, are desirably removed as a cohesive unit to provide a clean, bare optical glass fiber which is substantially free of residue. This residue can interfere with the optical glass fiber ribbon mass fusion splicing operation, and therefore usually must be removed by wiping prior to splicing. However, the step of removing the residue can cause abrasion sites on the bare optical glass fiber, thus compromising the strength of the connection. The superior stripping functionality of ribbon assemblies to provide clean, residue-free, bare optical glass fibers during ribbon stripping according to this invention has heretofore been believed to be unobtainable.

A common method for practicing ribbon stripping at a terminus of the ribbon assembly is to use a heated stripping tool. Such a tool consists of two plates provided with heating means for heating the plates to about 90 to about 120 C. An end section of the ribbon assembly is pinched between the two heated plates and the heat of the tool softens the matrix material and the primary coatings on the individual optical glass fiber. The heat-softened matrix material and heat-softened primary coatings present on the individual optical glass fibers can then be removed to provide bare optical glass fiber ends, at which the fusion connections can be made. A knife cut is often used to initiate a break in the matrix material to the inner primary coating. Typically, only about a 1 to 4 cm section of the matrix material and coatings on the optical glass fibers need be removed. Identification of the bare individual optical glass fibers achieved by tracing back along the bare optical fiber until the ink coating or colored outer primary coating is seen.

U.S. Pat. No. 5,373,578 discloses a ribbon assembly containing a plurality of coated optical glass fibers. Each of the optical glass fibers is coated with an inner primary coating which is adjacent to the optical glass fiber, with an outer primary coating and an ink coating on the outer primary coating. The inner primary coating is modified so that adhesion between the inner primary coating and the optical glass fiber is reduced. This reduction in adhesion facilitates easy removal of the heat-softened primary coating when using a heat stripping method. While this patent discloses, at column 5, lines 10-13, that the adhesion between the inner primary coating and the optical glass fiber should be sufficient to prevent delamination of the inner primary coating from the optical glass fiber, any reduction in the adhesion between the inner primary coating and the optical glass fiber increases the possibility of such undesirable delamination, especially in the presence of moisture. Delamination of the inner primary coating from the optical glass fiber can lead to degraded strength of the optical glass fiber as well as signal transmission attenuation disadvantages.

Published European patent application 0262340 discloses a ribbon cable having a "peel layer" as the outermost coating layer on each of optical glass fibers contained within the ribbon cable. During ribbon stripping, the peel layer is destroyed and the matrix material is removed from the coated optical glass fibers. However, after ribbon stripping, the optical glass fibers are still coated with the primary coatings. That is, the primary coatings are not simultaneously removed with the matrix material in the ribbon assemblies disclosed in this publication.

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U.S. Pat. No. 5,011,260 discloses a ribbon cable having a “decoupling layer” disposed between the coated optical glass it fibers and the matrix material. In this manner, the matrix material may be easily removed from the coated optical glass fibers by application of low stripping force. This patent includes a general statement that the coatings on the optical glass fiber can be simultaneously removed with the matrix material during ribbon stripping. However, this patent fails to teach how to solve the problems associated with the residues remaining on the bare optical glass fibers after ribbon stripping conventional ribbon assemblies.

Published European patent application 0407004 discloses a ribbon cable containing a matrix material having sufficient adhesion to the ink coated optical glass fibers to remain adhered thereto during normal use but is easily strippable therefrom without damaging the integrity of the ink layer on the coated optical glass fibers. Thus, the ribbon assembly disclosed in this publication does not have the capability of removing the primary coatings on the optical glass fibers simultaneously with removal of the matrix material during ribbon stripping, so as to provide residue-free bare optical glass fibers.

Published European patent application 0527266 discloses a ribbon cable containing a lubricating “interfacial layer” which separates the matrix material from the coated optical glass fibers. The interfacial layer facilitates easy removal of the matrix material from the coated optical glass fibers. While this publication discloses at page 3, line 15, that the buffer layer and first protective coating can be stripped in one step, there is no disclosure teaching how to accomplish such an operation. Furthermore, the lubricating interfacial layer will inhibit simultaneous removal of the first protective coating with the matrix material. Thus, this publication does not teach how to make a ribbon assembly having the capability of removing the primary coatings on the optical glass fibers simultaneously with the matrix material during ribbon stripping, so as to provide residue-free bare optical glass fibers.

U.S. Pat. No. 4,900,126 discloses a ribbon cable in which the bonding adhesive forces between the ink layer and the primary coatings on the optical glass fibers are greater than the bonding between the ink layer and the matrix material. In this manner, the matrix material can be easily removed from the ink coated optical glass fibers without removing the ink layer. However, this patent does not address the problems associated with removing the primary coating layers simultaneously with the matrix material.

U.S. Pat. No. 4,660,927 teaches a silicone-coated optical fiber in which the soft silicone coating is easily peeled from the surface of the optical glass fibers by finger pressure. The coating contains a first siloxane component having aliphatic unsaturated groups and a second siloxane component having mercaptoalkyl groups. Because such a coating is easily peelable, as by rubbing with finger pressure, the coating has insufficient adhesion to the surface of the optical glass fibers to prevent delamination during most uses. Furthermore, this patent does not address the problems of ribbon stripping, but rather only the stripping of a single optical glass fiber. It is generally known that three coating systems (inner primary coating, outer primary coating, and ink coating) having acceptable single fiber strippability will exhibit dramatically different levels of strippability characteristics when used in ribbon form.

U.S. Pat. No. 4,496,210 provides a radiation-curable optical fiber coating composition containing a polysiloxane. However, this patent does not address the problems associated with ribbon stripping.

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Japanese Patent Application H3-35210 teaches to combine a liquid lubricant, such as liquid silicone oil or liquid aliphatic oil, with a mercaptosilane compound in an inner primary coating composition. During stripping, when the bond between the surface of the optical glass fiber and inner primary coating is broken the liquid lubricant invades the boundary between the surface of the optical glass fiber and the inner primary coating. The liquid lubricant must not have a high compatibility with the inner primary coating or it will not bleed out of the inner primary coating during stripping. However, this document fails to teach a system to adjust the level of fiber friction between the adjacent surfaces of the optical glass fiber and the inner primary coating to a level which provides a resistive force that is less than the cohesive strength of the inner primary coating. Thus, while this document teaches that the inner primary coating can be stripped more easily by incorporating liquid lubricant compounds, the inner primary coating will still leave unwanted residue on the surface of the optical glass fiber if the above described fiber friction forces are at a level which provide a resistive force that is greater than the cohesive strength of the inner primary coating.

One primary coating composition available from JSR Corporation, designated as R-1055, is specified as having, inter alia, a viscosity of 5000 cps @25° C., a glass transition temperature of -4° C., a shrinkage value of 2.9%, a tensile strength value of 0.21 kg/mm<sup>2</sup>, a tensile elongation value of 195%, an adhesion force of 20 g/cm and a Young's modulus @23° C. of 0.12 kg/mm<sup>2</sup>. When this composition was tested in accordance with the test methods herein, it had a measured crack propagation value of 1.56 mm (standard deviation 0.2), and a fiber pull-out friction value of 26.3 g/mm (standard deviation 1.65).

There are many test methods which may be used to determine the performance of a ribbon assembly during ribbon stripping. An example of a suitable test method for determining the stripping performance of a ribbon is disclosed in the article by Mills, G., “Testing of 4- and 8-fiber ribbon strippability”, 472 International Wire & Cable Symposium Proceedings (1992), the complete disclosure of which is incorporated herein by reference.

Many attempts have been made to understand the problems associated with ribbon stripping and to find a solution to increase ribbon stripping performance. The following publications attempt to explain and solve the problems associated with ribbon stripping: K. W. Jackson, et. al., “The Effect of Fiber Ribbon Component Materials on Mechanical and Environmental Performance”, 28 International Wire & Symposium Proceedings (1993); H. C. Chandon, et. al., “Fiber Protective Design for Evolving Telecommunication Applications”, International Wire & Symposium Proceedings (1992); J. R. Toler, et. al., “Factors Affecting Mechanical Stripping of Polymer Coatings From Optical Fibers”, International Wire & Cable Symposium Proceedings (1989); and W. Griffioen, “Strippability of Optical Fibers”, EFOC & N, Eleventh Annual Conference, Hague (1993).

The ability of a ribbon assembly to ribbon strip cleanly so as to provide bare optical glass fibers that are substantially free of residue is still unpredictable and the factors affecting ribbon stripping are not fully understood. There is still a need for an understanding of how the problems of ribbon stripping occur and a solution to these problems.

#### SUMMARY OF THE INVENTION

It is an objective of the present invention to provide a novel ribbon assembly having improved ribbon stripping



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capabilities. It is another objective of the present invention to provide a novel ribbon assembly which after ribbon stripping provides bare optical glass fibers which are substantially free of residue, that must be removed prior to forming connections to the respective selected bare optical fibers.

Surprisingly, the above objects and other objects are and have been obtained by the following. The present invention provides a novel ribbon assembly comprising:

- a plurality of coated optical glass fibers, at least one optical glass fiber coated with at least an inner primary coating and an outer primary coating, and optionally an ink coating; and
- a matrix material bonding said plurality of coated optical glass fibers together, wherein said inner primary coating is adapted to provide the combination of properties of:
  - (i) sufficient adhesion to said optical glass fiber to prevent delamination during handling and in the presence of moisture; and
  - (ii) a fiber friction force between said optical glass fiber and said inner primary coating which has been so adjusted as to allow the inner primary coating to slide readily off from the optical glass fiber while leaving substantially no residue on the surface of said optical glass fiber during ribbon stripping, when a stripping force which is less than the cohesive strength of said inner primary coating is applied to said ribbon assembly.

Also provided is a novel ribbon assembly comprising:

- a plurality of optical glass fibers, at least one coated optical glass fiber coated with at least an inner primary coating and an outer primary coating, and optionally an ink coating; and
  - a matrix material bonding said coated optical glass fibers together,
- and wherein said inner primary coating is adapted to provide a fiber pull-out friction of about 30 grams/millimeter or less at a rate of about 0.1 mm/sec in combination with a crack propagation characteristic of at least about 1 millimeter at a rate of 0.1 mm/sec.

The present invention further provides a coated optical glass fiber comprising:

- an optical glass fiber;
- an inner primary coating on the surface of said optical glass fiber;
- an outer primary coating substantially co-extensive with the external surface of said inner primary coating, wherein said inner and outer primary coatings are so formulated and selected so as to provide a ratio of (i) the change in length of the inner primary coating from an ambient temperature to a ribbon stripping temperature to (ii) the change in length of the outer primary coating from said ambient temperature to said ribbon stripping temperature of less than about 1.5:1; and
- optionally an ink coating adjacent to said outer primary coating.

The invention further relates to a ribbon assembly containing at least one of these coated optical glass fibers.

The present invention further relates to a novel radiation-curable oligomer which can be used to adjust the fiber friction to a level such that the resulting adhesive resistive force level is less than the cohesive strength of the inner primary coating. The novel radiation-curable oligomer comprises:

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- at least one glass coupling moiety;
- at least one slip agent moiety; and
- at least one radiation-curable moiety, wherein said glass coupling, glass adhesion, and radiation curable moieties are each covalently linked to said oligomer.

Also provided is a radiation-curable, inner primary coating composition containing the composite oligomer, a coated optical glass fiber made from the coating composition, and a ribbon assembly containing at least one such coated optical glass fiber.

The present invention also provides a radiation-curable, inner primary coating composition comprising at least one radiation-curable oligomer or monomer and a wax. Preferably, the wax is present in an amount sufficient to provide a fiber friction between an inner primary coating formed from said coating and an optical glass fiber such that there is exhibited a resistive force that is less than the cohesive strength of said coating formed from said composition. The invention also provides a coated optical glass fiber having an inner primary coating which contains a wax, and a ribbon assembly which contains at least one such coated optical glass fiber.

The present invention further provides a coated optical glass fiber having an inner primary coating which has been formulated from a radiation-curable, inner primary coating composition containing a radiation-curable silicone oligomer or a silicone compound. Preferably, the radiation-curable silicone oligomer or silicone compound is present in an amount sufficient to provide a fiber friction between the inner primary coating and the optical glass fiber such that there is exhibited a resistive force which is less than the cohesive strength of the inner primary coating. The invention also provides a ribbon assembly which contains at least one such coated optical glass fiber.

The present invention also provides a coated optical glass fiber having an inner primary coating which has been formulated from a radiation-curable, inner primary coating composition containing a radiation-curable fluorinated oligomer or a fluorinated compound. Preferably, the radiation-curable fluorinated oligomer or fluorinated compound is present in an amount sufficient to provide a fiber friction between the inner primary coating and the optical glass fiber such that there is exhibited a resistive force that is less than the cohesive strength of the inner primary coating. The invention further provides a ribbon assembly which contains at least one such coated optical glass fiber.

The present invention also provides a radiation-curable, inner primary coating composition comprising at least one radiation-curable oligomer or monomer and a solid lubricant which is substantially insoluble in the composition. Preferably, the solid lubricant is present in an amount sufficient to provide a fiber friction between an inner primary coating formed from said coating and an optical glass fiber such that there is exhibited a resistive force which is less than the cohesive strength of said coating formed from said composition. The invention also provides a coated optical glass fiber having an inner primary coating which contains a solid lubricant, and a ribbon assembly which contains at least one such coated optical glass fiber.

The present invention further provides a ribbon assembly comprising a plurality of coated optical glass fibers, at least one optical glass fiber coated with at least an inner primary coating and an outer primary coating, and optionally an ink coating, and a matrix material bonding said plurality of coated optical glass fibers together. The inner primary coating is formulated from a radiation-curable inner primary coating composition containing at least one radiation-

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curable urethane oligomer comprising at least one polymeric block and at least one functional group capable of polymerization in the presence of actinic radiation connected to said at least one polymeric block. The coating composition has a concentration of urethane groups which is selected to provide said inner primary coating with a fiber friction force level between said optical glass fiber and said inner primary coating in combination with a crack propagation level that provides the inner primary coating with the functional capability of sliding off of the optical glass fiber and leaving substantially no residue on the surface of said optical glass fiber during ribbon stripping when a stripping force which is less than the cohesive strength of said inner primary coating is applied to said ribbon assembly.

The present invention further provides a ribbon assembly comprising a plurality of coated optical glass fibers, at least one optical glass fiber coated with at least an inner primary coating and an outer primary coating, and optionally an ink coating, and a matrix material bonding said plurality of coated optical glass fibers together. The inner primary coating is formulated from a radiation-curable inner primary coating composition containing at least one radiation-curable oligomer comprising at least one polymeric block and at least one functional group capable of polymerization in the presence of actinic radiation connected to said at least one polymeric block. The polymeric block has a molecular weight which is selected to provide said inner primary coating with a fiber friction force level between said optical glass fiber and said inner primary coating in combination with a crack propagation level that provides the inner primary coating with the functional capability of sliding off of the optical glass fiber and leaving substantially no residue on the surface of said optical glass fiber during ribbon stripping when a stripping force which is less than the cohesive strength of said inner primary coating is applied to said ribbon assembly.

The invention also provides a radiation-curable, inner primary coating composition formulated from a composition comprising at least one urethane oligomer having at least one polymeric block and at least one functional group capable of polymerization in the presence of actinic radiation connected to said at least one polymeric block. The coating composition has a concentration of urethane groups that is so selected to provide said inner primary coating with a fiber friction force level between an optical glass fiber and an inner primary coating formed from said coating composition in combination with a crack propagation level which provides the inner primary coating with the functional capability of sliding off the optical glass fiber and leaving substantially no residue on the surface of said optical glass fiber during ribbon stripping when a stripping force which is less than the cohesive strength of said inner primary coating is applied to said inner primary coating.

The present invention further provides a radiation-curable, inner primary optical glass fiber coating composition formulated from a composition comprising at least one radiation-curable oligomer having at least one polymeric block and at least one functional group capable of polymerization in the presence of actinic radiation connected to said at least one polymeric block. The polymeric block has a molecular weight so selected to provide said inner primary coating with a fiber friction force level between said optical glass fiber and said inner primary coating in combination with a crack propagation level that provides the inner primary coating with the functional capability of sliding off the optical glass fiber and leaving substantially no residue on the surface of said optical glass fiber during ribbon stripping

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when a stripping force which is less than the cohesive strength of said inner primary coating is applied to said inner primary coating.

The present invention also provides coated optical glass fibers containing at least one inner primary coating formed from the above radiation-curable, inner primary coating compositions.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates a longitudinal cross-sectional view of a coated optical glass fiber.

FIG. 2 illustrates a representative graph of the normalized strip force required to slide an optical fiber ribbon coating composite along the surface of an optical glass fiber.

FIG. 3 illustrates the ratchet effect of an inner primary coating sliding off an optical glass fiber during ribbon stripping.

FIG. 4 is a graph of the change in length  $L$  ("dL") for a commercially available outer primary coating as the temperature is increased.

FIG. 5 illustrates a partial cross-sectional view of a coated optical glass fiber.

FIG. 6 illustrates a hypothetical contour plot for determining the predicted strip cleanliness.

FIG. 7 illustrates a graph of the fiber pull-out friction versus urethane concentration.

FIG. 8 illustrates a graph of the fiber pull-out friction versus urethane concentration.

FIG. 9 illustrates a graph of the fiber pull-out friction versus urethane concentration.

FIG. 10 illustrates a graph of the fiber pull-out friction versus urethane concentration.

FIG. 11 illustrates a graph of the fiber pull-out friction versus urethane concentration.

#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

The invention will now be explained in detail with reference to the attached drawings.

Based on extensive experimentation, it is now believed that ribbon stripping functionally involves two phases, a first adhesion breaking phase and a second frictional sliding phase. This can be characterized by the following equation (1):

$$F_{\text{stripping}} = F_{\text{adhesive}} + F_{\text{friction}} \quad (1)$$

where

$F_{\text{stripping}}$  is the stripping force applied to the inner primary coating;

$F_{\text{adhesive}}$  is the force required to break the adhesive forces between the optical glass fiber and the inner primary coating; and

$F_{\text{friction}}$  is a function of the normal force the inner primary coating exerts against the surface of the optical glass fiber and the coefficient of friction of the inner primary coating.

$F_{\text{friction}}$  is equal to  $F_{\text{static}}$ , defined as the condition when the inner primary coating is in a static position, and  $F_{\text{friction}}$  is equal to  $F_{\text{kinetic}}$  defined as when the inner primary coating is in motion relative to the optical glass fiber.

During the adhesion breaking phase, the adhesive force between the inner primary coating and the surface of the optical glass fiber must be broken to delaminate the inner primary coating from the surface of the optical glass fiber.

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Once that adhesive force is broken, and the inner primary coating is delaminated from the surface of the optical glass fiber, the fiber friction force between the inner primary coating and the surface of the optical glass fiber must then be overcome to remove the inner and outer primary coatings, along with the matrix material, from the optical glass fiber.

The adhesive force between the inner primary coating and the surface of the optical glass fiber is generally increased by an increase in the following:

- (1) covalent bonding, for example from glass adhesion promoters;
- (2) weak molecular interactions, such as Van der Waal's attractions, hydrogen-bonding, electrostatic, and the like;
- (3) static coefficient of friction;
- (4) surface energy of the inner primary coating and surface energy of the optical glass fiber;
- (5) surface roughness; and
- (6) adhesive bonding area.

The adhesive force between the inner primary coating and the surface of the optical glass fiber is generally decreased by an increase in the temperature.

The fiber friction force between the inner primary coating and the surface of the optical glass is generally increased by an increase in one or more of the following:

- (1) the normal force of the inner primary coating against the surface of the optical glass fiber at the ribbon stripping temperature;
- (2) the static and kinetic coefficient of friction at the ribbon stripping temperature;
- (3) surface roughness; and
- (4) frictive area.

The normal force includes weak molecular interactions, such as Van der Waal's attractions, hydrogen-bonding, electrostatic, and the like, between the surface of the optical glass fiber and the inner primary coating. In general, the fiber friction force is decreased with an increase in temperature.

The rigidity and integrity of the outer primary coating at the ribbon stripping temperature can also affect the frictive force. During ribbon stripping the outer primary coating, ink coating, and other rigid coating layers, such as the matrix material, provide the stiffening backbone which allows for intact removal of the matrix material and inner and outer primary coatings to provide a cohesive tube (hereinafter referred to as "coating tube"). If the rigidity and integrity are insufficient, the outer primary coating can buckle during ribbon stripping, which can significantly increase the fiber friction force and/or induce shearing stresses causing integrity failure of the inner primary coating resulting in undesirable residue on the surface of the optical glass fiber.

Preferably, the adhesion between the matrix material and the ink coating or colored outer primary coating is greater than the adhesion between the inner primary coating and the surface of the optical glass fiber to ensure that the inner primary coating delaminates from the surface of the optical glass fiber during ribbon stripping. Similarly, both the adhesion between the ink coating and the outer primary coating, and the adhesion between outer primary coating and the inner primary coating, should be greater than the adhesion between the inner primary coating and the surface of the optical glass fiber to ensure that the inner primary coating delaminates from the surface of the optical glass fiber, as well as to provide a cohesive coating tube during ribbon stripping. Usually, the adhesion between the matrix material and the colored outer primary coating or ink coating, as well

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as the adhesion between each of the coating layers is sufficient to ensure delamination of the inner primary coating from the surface of the optical glass fiber during ribbon stripping because the matrix material and coating layers mainly comprise organic materials. In general, layers of materials having similar properties, such as an adjacent organic layer/organic layer bond, tend to bond more easily together than layers having dissimilar properties, such as an organic layer/inorganic layer bond.

FIG. 1 illustrates an optical glass fiber 7 coated with an inner primary coating 8 and a commercially available outer primary coating 9. The length of the inner primary coating in FIG. 1 shown at 20 has been selected to be 35 mm because this is a typical length of the coatings stripped from the ends of the optical glass fibers during ribbon stripping. When a typical ribbon stripping tool is applied to a ribbon assembly, pressure is applied to the ribbon assembly between heated plates. At the ends of the plates near the cut made in the matrix material and inner and outer primary coatings, the inner primary coating can form an initial delamination site on the optical glass fiber, shown at 27 and 28 (referred to as debond area). Because the areas 27 and 28 of the inner primary coating are delaminated, they must be subtracted from that area of the inner primary coating which is still bonded to the surface of the optical glass fiber when measuring the adhesive bonding area between the inner primary coating and the surface of the optical glass fiber. The radius of the optical glass fiber is 62.5 microns, shown at 22. The radius of the outer surface of inner primary coating is 95 microns, shown at 24. The radius of the outer surface of the outer primary coating is 125 microns, shown at 26. From FIG. 1, the adhesive bonding area is equal to the glass surface area (13.744 mm<sup>2</sup>) minus the debond area. The frictive area is the total glass surface area of the section to be stripped during ribbon stripping (13.744 mm<sup>2</sup>).

It is believed that during ribbon stripping the inner primary coating may ratchet off the optical glass fiber, as shown in FIG. 2. FIG. 2 is a demonstration of the adhesive force and fiber friction force being overcome by the stripping force applied to the inner primary coating during the ribbon stripping process. As stripping force is applied from the stripping tool to the inner primary coating, the stripping force increases to a level at which the adhesive force between the inner primary coating and the surface of the optical glass fiber is overcome, which is shown at 1. At this level, the inner primary coating begins to delaminate from the surface of the optical glass fiber. Then, the stripping force decreases as the inner primary coating delaminates from the optical glass fiber, shown generally at 2. Once delamination has completed, shown at 3, the inner primary coating slides along the surface of the optical glass fiber and the stripping force decreases to the level shown at 4. As the inner primary coating is being slid off of the optical glass fiber the stripping force required to slide the inner primary coating against the optical glass fiber ratchets between the higher static fiber friction force and the lower kinetic fiber friction force.

The static fiber friction force is a function of the static coefficient of friction of the inner primary coating and the normal force of the inner primary coating against the optical glass fiber. The kinetic fiber friction force is a function of the kinetic coefficient of friction of the inner primary coating and the normal force of the inner primary coating against the optical glass fiber. The static fiber friction force resists initial sliding movement and the kinetic fiber friction force resists subsequent sliding movement. In other words, once the adhesive bond is broken and the static fiber friction force is

overcome, shown at 3, the inner primary coating slides a set distance until the kinetic fiber friction force prevents further motion and the inner primary coating becomes momentarily stuck in place against the surface of the optical glass fiber, shown at 4. As the stripping force increases, and before the inner primary coating resumes its motion relative to the optical glass fiber, the potential energy is stored in the inner primary coating which produces a tensile force and a stripping force within the inner primary coating. The tensile force is opposed to the normal force and the stripping force is opposed to the fiber friction force.

The motion force ("F<sub>motion</sub>") of the inner primary coating is a vector sum of the tensile force ("F<sub>tensile</sub>") and the stripping force ("F<sub>stripping</sub>"). The resistive force ("F<sub>resistive</sub>") is a vector sum of the fiber friction force ("F<sub>friction</sub>") and the normal force ("F<sub>normal</sub>") on the inner primary coating against the surface of the optical glass fiber.

Once the motion force ("F<sub>motion</sub>") exceeds the resistive force ("F<sub>resistive</sub>"), the inner primary coating begins to slide, shown at 5. The inner primary coating quickly slides a set distance and then becomes momentarily stuck, shown at 6. The distance the inner primary coating slides along the surface of the optical glass fiber between the points 5 and 6 in FIG. 2 is referred to as slip-stick distance. The slip-stick distance will vary and be dependent upon the materials used in the inner primary coating and optical glass fiber, and will also be dependent upon random probability due to non-homogeneity in the inner primary coating and optical glass fiber surface.

FIG. 3 further explains the ratcheting effect during ribbon stripping by way of example. As shown in FIG. 3, a partial longitudinal cross-section of an optical glass fiber is shown at 7. A two-dimensional vector explanation will be used herein for ease of explanation. However, it is understood that a coated optical glass fiber is a three-dimensional object and all of the described vectors need to be extended an additional dimension.

The optical glass fiber is coated with an inner primary coating shown at 8, and an outer primary coating shown at 9. The thickness "Y" of the inner primary coating is about 37.5 microns, shown at 12. As stripping force is indirectly applied to the inner primary coating in the direction shown at 10, the inner primary coating is deformed a pre-slip distance "X", shown at 11, at which point the inner primary coating delaminates and begins to ratchet along the surface of the optical glass fiber. The stripping force required to make the inner primary coating begin to ratchet along the surface of the optical glass fiber can be calculated as follows. The length of the tensile deformation of the deformed inner primary coating at the level of strip force required to make the inner primary coating begin to slide after being momentarily stuck to the surface of the optical glass fiber "Z" is shown at 13. The % elongation of the deformed inner primary coating can be calculated from the values Z and Y using the following equation (2):

(Z-Y)/Y=% elongation (2)

From a stress/strain curve, one skilled in the art can readily use the % elongation to calculate the tensile force (F<sub>tensile</sub>) required to initiate sliding of the inner primary coating from a static position.

The vector for the static fiber friction force F<sub>friction</sub> is shown at 19. When F<sub>motion</sub> is greater than F<sub>resistive</sub>, the inner primary coating will begin to slide from a static position. F<sub>motion</sub>, shown at 15, is the vector sum of F<sub>tensile</sub>, shown at 14, and F<sub>stripping</sub>, shown at 16. F<sub>resistive</sub>, shown at 17, is the vector sum of F<sub>friction</sub>, shown at 19, and F<sub>normal</sub>, shown at 18.

If either of the vector components F<sub>stripping</sub> or F<sub>tensile</sub> is greater than the corresponding inner primary coating resistive vector components (shear strength and tensile strength, respectively), then the inner primary coating will cohesively fail during ribbon stripping leaving an undesirable residue of inner primary coating material on the surface of the optical glass fiber.

Similarly, if either of the vector components F<sub>friction</sub> or F<sub>normal</sub> is greater than the inner primary coating resistive vector components (shear strength and tensile strength, respectively), then the inner primary coating will cohesively fail during ribbon stripping leaving an undesirable residue of inner primary coating material on the surface of the optical glass fiber.

More generally, the inner primary coating will cohesively fail if F<sub>resistive</sub> is greater than the cohesive strength of the inner primary coating. Thus, to prevent such residue, the F<sub>friction</sub> and/or F<sub>normal</sub> should be so adjusted as to provide a F<sub>resistive</sub> that is less than the cohesive strength of the inner primary coating.

The term "cohesive strength" of the inner primary coating is used herein to mean the amount of force necessary to destroy the integrity of the inner primary coating. Thus, a higher cohesive strength will require a greater amount of force to destroy the integrity of the inner primary coating. The cohesive strength can be measured using any one of (1) the shear strength of the inner primary coating, (2) the tensile strength of inner primary coating, or (3) the crack propagation of the inner primary coating. Preferably, the cohesive strength is measured using the crack propagation test, as described herein below.

This residue can interfere with the optical glass fiber ribbon mass fusion splicing operation, and therefore must be removed prior to splicing by wiping. The step of removing the residue can cause abrasion sites on the bare optical glass fiber, thus compromising the strength of the connection.

Once the adhesive bonds have been broken and the inner primary coating has been delaminated from the surface of the optical glass fiber, the ability of a ribbon assembly to strip cleanly during ribbon stripping and to provide bare optical glass fibers which are substantially free of residue can be understood using the following simplified equation (3):

F<sub>friction</sub>=C<sub>f</sub>F<sub>normal</sub> (3)

where

F<sub>friction</sub> is the static frictive force between the inner primary coating and the optical glass fiber;

C<sub>f</sub> is the static coefficient of friction of the inner primary coating on the surface of the optical glass fiber, and

F<sub>normal</sub> is the normal force of the inner primary coating against the surface of the optical glass fiber.

Hereinafter, the use of the term "fiber friction" in the specification and claims refers to the static fiber friction force.

In general, the lower the fiber friction, the lower the resistive force, and the easier the inner primary coating can be removed from the surface of the optical glass fiber without leaving a residue. From equation 3, it is evident that the fiber friction can be reduced by decreasing either or both the static coefficient of friction or the normal force.

Each inner primary coating has a specific cohesive strength which maintains the integrity of the inner primary coating. The greater the cohesive strength of the inner primary coating the greater the amount of energy required to break apart or fracture the inner primary coating. Thus, an

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inner primary coating having a higher cohesive strength can withstand greater stripping forces during ribbon stripping, without breaking apart and leaving residue on the surface of the optical glass fiber, than an inner primary coating having a lower cohesive strength.

From the above discussion, it is clear that if the fiber friction is at a level which provides a resistive force that is greater than the cohesive strength of the inner primary coating, then the inner primary coating will break apart leaving residue on the surface of the optical glass fiber. Thus, when selecting or formulating the inner and outer primary coatings, the fiber friction level should be adjusted taking into account the cohesive strength of the inner primary coating so that fiber friction provides resistive force that is less than the cohesive strength of the inner primary coating. Minimizing the Normal Force

From the above equations, the fiber friction force between the optical glass fiber and inner primary coating can be lowered by reducing the normal force of the inner primary coating against the surface of the optical glass fiber. In general, the greater the normal force, the greater the fiber friction force between the optical glass fiber and the inner primary coating. In other words, the harder the inner primary coating is pressing against the surface of the optical glass fiber, the harder it will be to slide the inner primary coating against the surface of the optical glass fiber and the greater the chances of leaving residue from the inner primary coating on the surface of the optical glass fiber. Since the normal force is a component of the fiber friction, lowering the normal force will lower the fiber friction. The normal force should therefore be adjusted or selected so as to provide a normal force vector component and a fiber friction vector component that provides a vector sum (resistive force) which is less than the cohesive strength of the inner primary coating.

During ribbon stripping, the inner primary and outer primary coatings are heated, typically to about 90 C. to about 120 C. Because inner primary coatings usually have a lower Tg than that of outer primary coatings, inner primary coatings usually expand to a greater extent than the outer primary coatings during ribbon stripping. Thus, when the inner and outer primary coatings are heated, the inner primary coating expands to a greater extent than the outer primary coating causing a pressure build-up within the inner primary coating and between the surface of the optical glass fiber and the outer primary coating. This pressure buildup in the inner primary coating increases the normal force of the inner primary coating against the optical glass fiber, thereby increasing the fiber friction force between the inner primary coating and the surface of the optical glass fiber. Thus, the resistive force will be increased by an increase in the normal force vector component and an increase in the fiber friction vector component.

It is believed that the inner primary coating expands to a greater extent than the outer primary coating during ribbon stripping, at least in part due to the following reason. At temperatures below the Tg of the polymeric coating, the polymers present in the coating tend to act "glass-like", and therefore have a low coefficient of expansion. However, at temperatures above the Tg of the polymeric coating, the polymers tend to act "rubber-like" and therefore have a higher coefficient of expansion than when below the Tg of the polymeric coating. As the temperature of the ribbon assembly is raised during ribbon stripping the polymer present in the inner primary coating will usually be at a temperature above their Tg and be more "rubber-like" well before the polymers present in the outer primary coating

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reach their Tg. Thus, as the applied stripping temperature is raised, the "rubber-like" polymer present in the inner primary coating will expand to a much greater extent, than the "glass-like" polymer in the outer primary coating.

The Tg of the inner primary coating and that of the outer primary coating usually cannot be matched because the outer primary coating should have a higher Tg to provide the tough protective properties required of the outer primary coating. In general, the Tg of the outer primary coating is above 60° C., whereas the Tg of the inner primary coating is usually below 10° C., preferably below about 0° C., more preferably below about -10° C., and most preferably below about -20° C.

However, it has been found that the relative expansion characteristics of the inner and outer primary coatings can be adjusted without substantially affecting the Tg of the coatings. The expansion characteristics of the desired inner and outer primary coatings should first be measured as follows. The change in expansion from the ambient working temperature of the ribbon assembly to the ribbon stripping temperature measured in one plane "dL" is divided by an initial length of the one plane measured at the ambient working temperature of the ribbon assembly "L", hereinafter referred to as "(dL/L)". Ambient working temperatures of ribbon assemblies are usually about 0° C. to about 30° C. It will be appreciated that for most coating compositions the design ribbon stripping temperatures are usually about 90° C. to about 120° C., but may be different depending on the specific design parameters for the particular coating composition.

The inner and/or outer primary coatings should be selected or reformulated so as to maximize the (dL/L) of the outer primary coating and while minimizing the (dL/L) of the inner primary coating. Ideally, the (dL/L) of the outer primary coating should be greater than that of the inner primary coating whereby the outer primary coating will theoretically exert a normal force on the inner primary coating in a direction away from the optical glass fiber during ribbon stripping. However, such high (dL/L) values for the outer primary coating in combination with retention of the desired toughness properties of the outer primary coating are usually unattainable. Nevertheless, increasing the (dL/L) of the outer primary coating can significantly reduce the increase in normal force on the inner primary coating during ribbon stripping to provide a clean optical glass fiber which is substantially free of residue.

FIG. 4 is a graph of the change in L ("dL") for a commercially available outer primary coating as the temperature is increased. In particular, for an L of 23.2 mm, the dL for a temperature change from 25 C. (example of ambient temperature) to 100 C. (example of ribbon stripping temperature) can be calculated as follows:

$$\begin{aligned} dL/L &= (\text{delta } L)/L \\ &= (0.4)/23.2 \\ &= .01724 \end{aligned}$$

The dL/L value is independent of the length of the coating selected for the measurement. Thus, for different L values, the dL/L will be constant.

The normal force on the inner primary coating against the optical glass fiber, which is caused by the differential in expansion between the inner primary coating and outer primary coating during ribbon stripping, can be calculated as follows. FIG. 5 illustrates a cross-sectional view of a glass optical fiber 7, coated with an inner primary coating 8 and

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an outer primary coating 9. The outer primary coating 9 is the same as that in FIGS. 1 and 4. The radius of the outer surface of the inner primary coating at 25 C. is 95 microns, shown at 40. The radius of the inner surface of the outer primary coating at 25 C. is 95 microns, also shown at 40. As the temperature of the ribbon assembly is increased to 100 C. during ribbon stripping, the inner primary coating and outer primary coating expand.

The radius of the inner surface of the outer primary coating at 100 C. is 96.379 microns, shown at 42. This value was calculated as follows. The (dL/L) for the outer primary coating material heated from 25 C. to 100 C. is 0.01724, as calculated from FIG. 4. The radius of the inner surface of the outer primary coating at 25 C. (95 microns) is multiplied by (1+dL/L) for a temperature change of 25 C. to 100 C. (1.01724) which provides a radius of the inner surface of the outer primary coating at 100 C. of 96.638 microns. However, this value must be corrected to take into account the expansion in the thickness of the outer primary coating. The outer primary coating has a thickness of 30 microns at 25 C. To obtain the thickness at 100 C., the thickness at 25 C. (30 microns) is multiplied by (1+dL/L) for a temperature change of 25 C. to 100 C. (1.01724), which provides a thickness of 30.5172 microns. Thus, the thickness of the outer primary coating expands 0.5172 microns when heated from 25 C. to 100 C. One half of this expansion occurs in the direction of the inner primary coating. This assumes that the inner primary coating will not substantially resist the expansion of the outer primary. Thus, one half of 0.5172 must be subtracted from the value obtained above for the radius of inner surface of the outer primary coating at 100 C. (96.638 microns) to obtain a corrected value of 96.379 microns. The change in radius over the temperature change from 25 C. to 100 C. "dR" divided by the radius at 25 C. "R" is then calculated to provide the value (dR/R).

The above measurements can be performed on the inner primary coating selected to provide a value (dR/R) for the inner primary coating. The radius of the inner primary coating at 100 C. is shown at 44. The normal force on the inner primary coating against the optical glass fiber, which is caused by the differential in expansion between the inner primary coating and outer primary coating during ribbon stripping is shown at 46.

The % expansion of the inner primary coating can be calculated from the following:

$$((dR/R)_{\text{inner primary}} - (dR/R)_{\text{outer primary}}) \times 100\%$$

From a stress/strain curve, one skilled in the art can easily use the % expansion to calculate the pressure of the inner primary coating against the optical glass fiber, which is caused by the differential in expansion between the inner and outer primary coating during ribbon stripping. Multiplying the pressure by the surface area of the inner primary coating against the optical glass fiber provides the normal force of the inner primary coating against the surface of the optical glass fiber.

Preferably, the (dR/R)<sub>inner primary</sub> is decreased and/or the (dR/R)<sub>outer primary</sub> is increased to reduce the differential in expansion between the inner and outer primary coating during ribbon stripping, thereby reducing the normal force of the inner primary coating against the surface of the optical glass fiber.

Based on the above, it has been found that the normal force can be decreased by reducing the pressure increase of the inner primary coating during ribbon stripping, by reformulating the inner primary coating and/or outer primary coating to provide one or more of the following properties:

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- (1) decreasing the elastic modulus (at ribbon stripping temperature) of the outer primary coating so that it can stretch to a greater extent to relieve more of the pressure build-up of the inner primary coating during ribbon stripping,
- (2) increasing the (dL/L) of the outer primary coating so that the outer primary coating expands to a greater extent to allow for more expansion of the inner primary coating during ribbon stripping, and/or
- (3) decreasing the (dL/L) of the inner primary coating to reduce the pressure build-up of the inner primary coating.

The elastic modulus (at ribbon stripping temperature) of the outer primary coating can be decreased by reducing the crosslink density of the outer primary coating. The elastic modulus is determined by the Elastic Modulus Test method, as discussed in the DESCRIPTION OF TEST METHODS, below. Preferably, the elastic modulus of the outer primary coating is adjusted to be between about 10 to about 40 MPa, more preferably between about 10 to about 20 MPa, at the ribbon stripping temperature. Outer primary coatings having an elastic modulus in the range of between about 15 to about 40 MPa, more preferably between about 30 and 40 MPa, have also been found suitable as well as outer primary coatings having an elastic modulus of greater than about 25 MPa. While it has been found that the crosslink density of the outer primary coating can usually be reduced without causing undesirable effects, the Tg of the outer primary coating should remain high, to provide the outer primary coating with the necessary toughness related properties to protect the optical glass fiber. For example, to reduce the crosslink density of the outer primary coating without reducing the Tg to unacceptably low values, monofunctional monomers or oligomers, which when cured exhibit a high Tg, can be used. Monofunctional is understood herein as including monomers and oligomers having an average of about 1 functional group capable of polymerization upon exposure to actinic radiation. A high Tg is herein understood to be at least about 40 C., preferably at least about 50 C.

Examples of suitable high Tg producing monofunctional monomers and oligomers include, for example, isobornyl acrylate and vinylcaprolactam. Such monomers can be utilized in amounts of about 1% to about 80%, preferably about 10 to about 50% by weight of the total composition.

Very high Tg producing multifunctional monomers or oligomers, such as tris-hydroxyethylisocyanurate triacrylate can also be used in amounts up to about 30%, preferably up to about 20% by weight, because they are effective at greatly increasing the Tg of the outer primary coating without excessively increasing the crosslink density.

The (dL/L) of the outer primary coating can be significantly increased by incorporating a monomer or oligomer which when cured exhibits a high (dL/L). For example, a suitable (dL/L), at the desired ribbon stripping temperature, for the outer primary coating has been found to be at least about 0.017, preferably at least about 0.02, and most preferably at least about 0.023. These amounts can be expressed as percentage increases in the length by multiplying by 100. Therefore, the outer primary coating preferably increases in length over the change in temperature from ambient temperature to ribbon stripping temperature ("dL/L") of at least about 1.7%, more preferably at least about 2%, and most preferably at least about 2.3%. If the coefficient of friction of the inner primary coating and/or the dL/L of the inner primary coating are sufficiently low enough, the dL/L of the outer primary coating can be less than 1.7% and still provide a fiber friction and normal force that will result in a resistive

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force that is less than the cohesive strength of the inner primary coating.

The high (dL/L) producing monomer or oligomer should be added in an amount sufficient to provide a cured outer primary coating with the desired level of (dL/L). For example, the high (dL/L) monomer or oligomer can be added in an amount of about 10 to about 70% by weight, more preferably about 10 to about 50% by weight.

Examples of suitable high (dL/L) monomers or oligomers include isobornyl acrylate, vinylcaprolactam, tricyclodecane dimethanol diacrylate, and the adduct of 2 moles of hydroxyethylacrylate and 1 mole of isophorone diisocyanate.

The (dL/L) of the inner primary coating can be decreased by increasing the crosslink density of the inner primary coating. However, when reformulating the inner primary coating to increase the crosslink density, the Tg of the inner primary coating should remain low to provide the optical glass fiber with adequate protection from microbending. It has been found that the crosslink density can be increased by using multifunctional monomers and oligomers. Examples of suitable multifunctional monomers and oligomers include, hexanedioldiacrylate, trimethylolpropane triacrylate, and tripropyleneglycol diacrylate.

The ratio of the dL/L (inner primary) to the dL/L (outer primary) at the desired ribbon stripping temperature should be low enough to provide a fiber friction and normal force that results in a resistive force between the inner primary coating and the optical glass fiber that is less than the cohesive strength of the inner primary coating. In general, the lower the ratio of dL/L (inner primary) to dL/L (outer primary) the less the normal force that will be applied to the inner primary coating against the surface of the optical glass fiber. Thus, the ratio of the dL/L (inner primary) to dL/L (outer primary) required to provide a fiber friction force that results in a resistive force lower than the cohesive strength of the inner primary coating will depend upon the coefficient of friction of the inner primary coating. The lower the coefficient of friction of the inner primary coating, the greater the ratio of dL/L (inner primary) to dL/L (outer primary) that can be tolerated and still provide a fiber friction and normal force which results in a resistive force that is less than the cohesive strength of the inner primary coating.

It has been found that a suitable ratio for the dL/L (inner primary) to the dL/L (outer primary) at the desired ribbon stripping temperature is less than about 2, preferably less than about 1.7, and most preferably less than about 1.5.

The outer primary coating can also exert a force on the inner primary, which is caused by shrinkage of the outer primary coating during radiation curing of the outer primary coating. Thus, to reduce this force oligomers and monomers can be selected to provide a radiation-curable composition that exhibits reduced shrinkage during radiation-curing.

If an ink coating is present, the ink coating can also exert a normal force on the inner primary coating in a manner similar to the normal force exerted by the outer primary coating. However, the force exerted by the ink coating will generally be significantly less than the force exerted by the outer primary coating because the ink coating is generally about an order of magnitude thinner than the outer primary coating. The thickness of the ink layer is usually only about 3 to about 8 microns.

If desired, the normal force exerted by the ink coating can be adjusted in a similar manner as adjusting the normal force exerted by the outer primary coating, because the ink coating in general is also formed from monomers and oligomers

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similar to those used to form the outer primary coating. In particular, the (dL/L) of the ink coating can be adjusted to be closer to the (dL/L) of the inner primary coating by reformulating the ink coating to utilize monomers and/or oligomers that result in a coating having a (dL/L) closer to the (dL/L) of the inner primary coating, as described herein above in reference to the outer primary coating.

The invention will be further explained by the following non-limiting example.

Comparative Examples A-1 to A-2

The compositions shown in Table 1 represent commercially available coating compositions. Comparative Example A-1 is an example of an outer primary coating and Comparative Example A-2 is an example of an inner primary coating.

TABLE 1

Component (Amount in % by weight of total composition)	Comp. Example A-1	Comp. Example A-2
Oligomer H-(T-PTMG650) <sub>1</sub> 14-T-H	39	
Oligomer H-(I-PTGL2000) <sub>2</sub> -I-H		51.41
Bisphenol A Diglycidylether	29	
Diacrylate		
Isobornyl Acrylate	10	6.86
Hexanediol Diacrylate	8.5	
Phenoxyethyl Acrylate	10	
Lauryl Acrylate		5.95
Ethoxylated Nonylphenol Acrylate		20.91
Tripropyleneglycol Diacrylate		5.81
Vinyl Caprolactam		6.11
Diethylamine		.1
Gamma Mercaptopropyl Trimethoxy Silane		1
Thiodiethylene bis(3,5-di-tert-butyl-4-hydroxy)hydrocinamate	.5	.31
2,4,6-Trimethylbenzoyl diphenyl phosphine oxide	2	1.54
1-Hydroxycyclohexylphenyl Ketone	1	

The oligomers were formed by reacting the following components:

H = Hydroxyethyl Acrylate

T = Toluene Diisocyanate

I = Isophorone Diisocyanate

PTGL2000 = 2000 molecular weight

polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl

copolymer diol (Mitsui, NY)

PTMG650 = 650 molecular weight polytetramethyleneglycol diol

(DuPont)

The compositions were suitably cured by exposure to UV light from a Fusion D lamp. The dL/L for each coating was measured over the temperature range of 25 C. (ambient temperature) to 125 C. (highest usual stripping temperature).

For Comparative Example A-1, the dL/L for a temperature change from ambient temperatures (25 C.) to ribbon stripping temperatures (100 C.) was 1.42%.

For Comparative Example A-2, the dL/L for a temperature change from ambient temperatures (25 C.) to ribbon stripping temperatures (100 C.) was 2.3%.

Thus, the ratio of the dL/L (inner primary) to dL/L (outer primary) was about 1.6, which would be acceptable if the coefficient of friction of the inner primary coating was low enough. However, the inner primary coating exhibited too high of a coefficient of friction, because the fiber friction estimated by using the fiber pull-out friction method described herein below was too great. The fiber pull-out friction was 39 g/mm, which resulted in a resistive force that was greater than the cohesive strength of the inner primary coating. Therefore, substantial amounts of inner primary coating residue were left on the optical glass fiber after ribbon stripping using the above inner and outer primary coatings.

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## Coefficient of Friction of Inner Primary Coating

From the above equations, fiber friction between the optical glass fiber and inner primary coating can also be adjusted by reducing the coefficient of friction of the inner primary coating against the surface of the optical glass fiber. By reducing the coefficient of friction of the inner primary coating, the "rubber-like" drag of the inner primary coating on the optical glass fiber is reduced.

Preferably, the coefficient of friction of the inner primary coating is reduced without reducing the adhesion of the inner primary coating to the surface of the optical glass fiber. If the adhesion is reduced, then undesirable delamination of inner primary coating from the surface of the optical glass fiber can occur.

It has been found that the fiber friction can be adequately adjusted to a value which provides resistive force below the cohesive strength of the inner primary coating by adjusting the coefficient of friction of the inner primary coating with the use of one or more of the novel slip additives described herein. Surprisingly, the coefficient of friction can be reduced to such a level without substantially reducing the adhesion of the inner primary coating to the optical glass fiber as follows.

Preferably, the ratio of the dL/L (inner primary) to the dL/L (outer primary) is adjusted in combination with adjusting the coefficient of friction of the inner primary coating to provide a fiber friction value which results in a resistive force that is less than the cohesive strength of the inner primary coating.

In practice, ribbon assemblies are generally stripped using a heated stripping tool. However, using the inventive concepts described herein, the present invention includes ribbon assemblies which surprisingly can be ribbon stripped at much lower temperatures, such as ambient temperatures, to provide bare optical glass fibers which are substantially free-of residue. It has been found that if the fiber friction between the inner primary coating and the surface of the optical glass fiber and/or the normal force is adjusted to a level which provides a resistive force lower than the cohesive strength of inner primary coating, the ribbon assembly will be ribbon strippable. Therefore, if a ribbon assembly which is adapted to provide ribbon strippability at ambient temperatures is desired, the fiber friction can be adjusted to a level to provide a resistive force that is less than the cohesive strength of the inner primary coating using slip agents as discussed herein. Alternatively, if the ribbon assembly is adapted to provide ribbon strippability at temperatures greater than ambient temperatures, the resistive force can be adjusted to a level lower than the cohesive strength of the inner primary coating by adjusting the ratio of the dL/L (inner primary) to the dL/L (outer primary) to provide a lower normal force and fiber friction, and/or adjusting the coefficient of friction of the inner primary coating using slip agents to provide a lower fiber friction.

## Novel Radiation-Curable, Silicone-Silane Oligomer

This invention also provides a novel type of radiation-curable oligomer that can be used to adjust the fiber friction between the inner primary coating and the surface of the optical glass fiber. The radiation-curable oligomer comprises a glass coupling moiety, a slip agent moiety, and a radiation curable moiety, each moiety being linked to a single composite oligomer molecule through covalent bonding to provide a composite oligomer. Such linkage of all three moieties is heretofore unknown. Linkage of these moieties can be direct so that no intermediate linking group between the oligomer and the moiety is required. Alternatively, however, the linkage can be indirect by using intermediate linking groups.

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A variety of glass coupling, slip agent, and radiation curable moieties are known in the art. The present invention can be practiced with use of various embodiments using different combinations of these moieties to produce a composite oligomer. A person skilled in the art will easily be able to prepare combinations of these various moieties from the present disclosure and general knowledge in the art.

Radiation-curing can occur by reaction of the composite oligomer's radiation-curable moieties with themselves or with radiation-curable moieties bound to other components of a formulation. In general, curing of the composite oligomer occurs in concert with other radiation-curable components. Radiation-curing, in the present invention, is associated with reaction of the radiation-curable moiety, not with the glass coupling or slip agent moieties. For example, although the glass coupling moiety will be reactive, and is often sensitive to hydrolysis and condensation reactions, these types of reactions are not the principal cure mechanism.

The molecular weight of the oligomer is not limited. In general, however, the molecular weight of the oligomer in its uncured state is usually between about 200 and about 10,000, preferably between about 500 and about 5,000. Molecular weight as used throughout this disclosure generally means number average molecular weight when measured, or the theoretical calculated molecular weight based on the reactants and reaction conditions used to make the composite oligomer.

There is no particular limitation on the molecular architecture of the composite oligomer, although in general, linear or substantially linear oligomeric structures are used rather than non-linear, cyclic, or branched structures. To the extent that the inventive concept can be practiced, however, branched or other non-linear structures are also envisioned and are not excluded. A substantially linear structure means that there is a single, dominant linear oligomeric backbone which is "capped" at the two ends of the backbone. The amount of branching units in the backbone is generally less than about 10 mole %, and preferably, less than about 5 mole %. The linear backbone may contain one or more types of repeat units, although preferably, one major type of repeat unit is used. Nevertheless, block or random copolymeric structures can be used if necessary. With a substantially linear backbone, the number of branch points in the backbone will be kept to a minimum, and preferably, will not be used. Synthetic simplicity in the oligomer structure is preferred to the extent that cost-performance can be achieved.

The term "glass coupling moiety" can be readily understood by a person skilled in the art and is understood to mean a functional group which is known or has the ability to improve adhesion to an inorganic surface or at an inorganic-organic interface, and in particular, a glass surface or at a glass-polymer interface. Such glass coupling moieties are associated with conventional coupling agents or adhesion promoters, as known to those skilled in the art. These conventional coupling agents generally have (1) an organic functional group which bonds with, or is at least associated with, the organic material at the interface, and (2) an inorganic component which bonds, usually covalently, to the inorganic material at the interface. Although the complexities of such bonding are not fully understood, usually, bonding to the inorganic surface occurs following hydrolysis and/or condensation reactions.

Exemplary conventional silane coupling agents are disclosed in E. P. Plueddemann's *Silane Coupling Agents*, Plenum Press (1982), the complete disclosure of which is hereby incorporated by reference. Non-silane types of cou-



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pling agents are also known and include, for example, chromium, orthosilicate, inorganic ester, titanium, and zirconium systems. Although the present invention is preferably practiced with use of silane glass coupling moieties, the invention is not so restricted, and a person skilled in the art is enabled by the present disclosure to use these other systems as well.

In the present invention, the glass coupling moieties are not part of a conventional coupling agent, but are incorporated covalently into the oligomer in a manner which preserves their coupling function to the inorganic surface or at the inorganic-organic interface. In a preferred embodiment, for example, the organic component of a conventional coupling agent is linked covalently, either directly or indirectly, with the composite oligomer together with the slip agent and radiation curable moieties. After this linkage, the glass coupling moiety will still have its inorganic component effective for bonding with the inorganic surface or at the inorganic-organic interface. However, the invention is not so limited, and the glass coupling moiety is not necessarily linked to the composite oligomer by reaction of the organic functional group of a conventional coupling agent.

Silane coupling moieties are preferred. These moieties can be created by covalently linking a conventional coupling agent or adhesion promoter with the oligomer. Representative types of silane coupling moieties have been disclosed in the aforementioned Plueddemann reference and the product information publication from Union Carbide entitled "UNION CARBIDE® Organofunctional Silanes Products and Applications" (1991, 1992), the complete disclosure of which is hereby incorporated by reference. The inorganic component of the conventional silane coupling agent is generally represented by the formula:



where

R is a conventional lower, and preferably, a C<sub>1</sub>-C<sub>4</sub>, alkyl group such as methyl or ethyl which imparts at least some hydrolyzability to the silane. Other types of R groups are also known in the art, however, and the invention is not particularly limited by the particular R group or silane structure to the extent that glass coupling can occur. Generally, at least one hydrolyzable " $-\text{Si}-\text{O}-\text{R}$ " linkage will be present in the glass coupling moiety to facilitate coupling to the surface of the optical glass fiber. Preferably, there is more than one such linkage. Hydrolyzable means that this linkage is sensitive to reaction with water to generate " $-\text{Si}-\text{OH}$ " linkages. In turn, " $-\text{Si}-\text{OH}$ " linkages are believed to condense to form " $-\text{Si}-\text{O}-\text{Si}-$ " linkages. In many cases, hydrolysis may even begin to occur with exposure to atmospheric moisture. Hydrolysis of silanes and glass surfaces in the context of optical fiber coatings is discussed in, for example: (i) the chapter entitled "Coating and Jackets", Chapter 10, Blyler et al. *Optical Fiber Telecommunications*, 1979, pgs. 299-341, and (ii) S. Wu, *Polymer Interface and Adhesion*, Marcel Dekker, 1982, pgs. 406-434, the complete disclosures of which are hereby incorporated by reference.

Common organic functionalities of the silane coupling agents include, for example, amino, epoxy, vinyl, methacryloxy, isocyanato, mercapto, polysulfide, and ureido. Using synthetic methods known in the art, the organic functionality can be reacted with the oligomer to yield a

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covalent linkage between the glass coupling moiety and the oligomer. In a preferred embodiment, for example, mercaptopropyl silane is linked with an oligomer containing an isocyanate group to form a thiourethane adduct between the mercapto group and the isocyanate group. Although a strong linkage is preferred, the present invention encompasses the possibility that although a covalent linkage is formed, the covalent linkage may not be strong and may, for example, be sensitive to disruption with the application of heat. However, as long as the glass coupling moiety produces the desirable effect of promoting adhesion, the covalent linkage is sufficient. If necessary, catalysts may be used to promote linkage formation.

Slip agents when used to practice this invention do not substantially affect the adhesion of the inner primary coating to the surface of the optical glass fiber. Instead, the slip agents reduce the sliding force of the inner primary coating against the surface of the optical glass fiber, once the bonds between the surface of the optical glass fiber and inner primary coating are broken (i.e. after the inner primary coating has been delaminated).

Slip agents are also known in the art as, among other things, release, antiblocking, antistick, and parting agents. Slip agents are commonly oligomeric or polymeric and are usually hydrophobic in nature, with the most common examples including silicones (or polysiloxanes), fluoropolymers, and polyolefins. If desired, the slip agent moiety can include silicones, fluoropolymers, and/or polyolefins in combination with polyesters, polyethers and polycarbonates. Slip agents are disclosed in, for example, the article entitled "Release Agents" published in the *Encyclopedia of Polymer Science*, 2nd Ed., Vol. 14, Wiley-Interscience, 1988, pgs. 411-421, the complete disclosure of which is hereby incorporated by reference. Although slip agents operate over a wide variety of interfaces, the present invention is particularly concerned with an interface of a glass surface, and in particular, a glass-organic coating interface between the inner primary coating and the surface of the optical glass fiber. A slip agent can be covalently incorporated into the composite oligomer as a slip agent moiety.

In a preferred embodiment, the slip agent moiety is the principal component of the oligomer in terms of weight percent because the slip agent moiety itself is usually oligomeric in nature, and the glass coupling and radiation-curable moieties are usually of lower molecular weight. For example, the slip moiety can be up to about 95 wt. % of the total composite oligomer weight when the three moieties are directly linked together. However, when an oligomeric backbone is present, the slip agent usually can be up to about 85 wt. % of the composite oligomer weight. As with the molecular weight of the composite oligomer of the present invention, the molecular weight of the slip agent moiety is not strictly limited, but will generally be between about 150 and about 9,500, preferably, between about 400 and about 4500.

As with the molecular architecture of the oligomer, there is no particular limitation on the molecular architecture of the slip agent moiety, although in general, substantially linear structures can be used. Non-linear or branched structures, however, are not excluded. Oligomeric slip agent moieties, when present, may contain different kinds of repeat units, although preferably, there is one main type of repeat unit.

Oligomeric silicone slip agent moieties are preferred, and oligomeric silicones comprising substantial portions of methyl side groups are particularly preferred. The side

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groups preferably impart hydrophobic character to the silicone. Other preferred side groups include ethyl, propyl, phenyl, ethoxy, or propoxy. In particular, dimethylsiloxane repeat units represented by the formula, " $-\text{OSi}(\text{CH}_3)_2-$ " are preferred.

In a preferred embodiment, the end groups on a substantially linear silicone oligomer can be linked with a radiation curable moiety at one end and a slip agent moiety at the other end. Such linkage can involve intermediate linkage groups. Although linkage at the silicone oligomer end group is preferred, the silicone moiety can be tailored for linkage with slip agent and radiation-curable moieties at other points in the oligomer molecule besides the end groups. For example, functional groups can be incorporated throughout the molecular structure of the silicone oligomer that are linked with the radiation-curable and slip agent moieties. Examples of functionalized silicones which can be incorporated into the oligomer include polyether, polyester, urethane, amino, and hydroxyl.

Other types of slip agent moieties including those made from fluorinated slip agents can also be used. Examples of suitable fluorinated slip agents include FC-430, FX-13, and FX-189 (Minnesota Mining and Manufacturing), Fluorolink E (Ausimont), and EM-6 (Elf Atochem).

Generally, the composite oligomer of the present invention is surface active because of the glass coupling moieties, and in particular, may tend to concentrate at coating interfaces, such as the glass-coating interface, if not bound in the inner primary coating. However, the covalent binding of the composite oligomer after cure, due to the radiation-curable moiety, may retard such surface activity or migration. Surface activity means that the composite oligomer, when placed in a formulation, tends to migrate to the surface of the formulation rather than be dispersed evenly throughout the formulation.

The radiation-curable moiety should help ensure that the composite oligomer is covalently linked within a radiation-curable coating so that the composite oligomer cannot be extracted or volatilized from the cured coating without breaking covalent bonds.

The radiation-curable moiety can include any functional group capable of polymerizing under the influence of, for example, ultraviolet or electron-beam radiation. One type of radiation-curable functionality is, for example, an ethylenic unsaturation, which in general is polymerized through radical polymerization, but can also be polymerized through cationic polymerization. Examples of suitable ethylenic unsaturation are groups containing acrylate, methacrylate, styrene, vinyl ether, vinyl ester, N-substituted acrylamide, N-vinyl amide, maleate esters and fumarate esters. Preferably, the ethylenic unsaturation is provided by a group containing acrylate, methacrylate or styrene functionality. Most preferably, the ethylenic unsaturation is provided by a group containing acrylate functionality.

Another type of functionality generally used is provided by, for example, epoxy groups, or thiol-ene or amine-ene systems. Epoxy groups, in general, can be polymerized through cationic polymerization, whereas the thiol-ene and amine-ene systems are usually polymerized through radical polymerization. The epoxy groups can be, for example, homopolymerized. In the thiol-ene and amine-ene systems, for example, polymerization can occur between a group containing allylic unsaturation and a group containing a tertiary amine or thiol.

The amount or number of glass coupling, slip agent, and radiation curable moieties in the composite oligomer is not particularly limited provided that advantages of the present

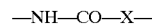
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invention can be achieved and the inventive concept is practiced. Thus, a single molecule of the composite oligomer can contain multiple numbers of glass coupling, slip agent, or radiation-curable moieties, although in a preferred embodiment, a single oligomeric molecule contains one glass coupling, one slip agent, and one radiation-curable moiety.

The glass coupling, slip agent, and radiation curable moieties should be covalently linked together in the oligomer. There is no particular limitation to how this linkage is effected provided that advantages of the present invention are achieved and the inventive concept practiced. Linkage may entail direct linkage to the oligomer, or alternatively, indirect linkage to the oligomer. Intermediate linking groups will generally operate by way of two functional groups on a linking compound which can link, for example, the radiation-curable moiety with the slip agent moiety, or link the glass coupling moiety with the slip agent moiety.

Representative linking compounds include diisocyanate compounds, wherein linkage occurs by formation of urethane, thiourethane, or urea links by reaction of hydroxyl, thiol, and amino groups respectively, with isocyanate. Such diisocyanate compounds are well-known in the polyurethane and radiation curable coating arts. Aromatic or aliphatic diisocyanates can be used, although aliphatic diisocyanates are preferred. Other linkages can be through, for example, carbonate, ether and ester groups. Preferably, urethane, urea or thiourethane groups are used as the linking groups.

The oligomer, therefore, preferably comprises within its structure at least one linkage represented by



wherein

X is an oxygen, sulfur, or nitrogen atom. Urethane and thiourethane groups are most preferred. Urethane groups, for example, can hydrogen bond.

Although the present invention is not limited to one particular molecular architecture for the composite oligomer, in a preferred embodiment which makes use of intermediate linking groups, the composite oligomer can be represented by the following generic structure:



wherein

A represents the slip agent moiety,

R represents a radiation-curable moiety,

C represents the glass coupling moiety, and

$\text{L}_1$  and  $\text{L}_2$  represent linking groups.

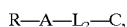
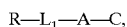
$\text{L}_1$  and  $\text{L}_2$  can be independently any group capable of providing a covalent link between the "R" moiety and the "A" moiety or between the "C" moiety and the "A" moiety. Based on the disclosure provided herein, one skilled in the art will easily be able to understand what linking groups are suitable for the particular "A", "C" and "R" groups selected.

In particular, urethane and thiourethane groups are preferred. Urethane and thiourethane linking groups are formed by, for example, (i) linking a hydroxyl end-capped oligomer with a low molecular weight diisocyanate compound at both oligomer ends without extensive coupling of the oligomer, (ii) linking the isocyanate end-capped oligomer with a low molecular weight hydroxyacrylate compound, or (iii) linking the isocyanate end-capped oligomer with a low molecular weight mercapto compound.

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The linking groups, however, are considered optional. In other words, the oligomer also can be represented by the following generic structures:



or



Although the present invention is disclosed in terms of the aforementioned groups or moieties, other groups can in principle be incorporated into the molecular structure to the extent that the advantages of the present invention can be achieved and the inventive concept practiced.

A preferred embodiment of the present invention is the preparation of a composite oligomer with use of the following ingredients: a silicone oligomer having two hydroxyl end groups (slip agent moiety), isophorone diisocyanate (linkage), hydroxyethyl acrylate (radiation-curable moiety), and mercaptopropyl silane (glass coupling moiety). isophorone diisocyanate (IPDI) serves to end-cap both ends of the silicone diol oligomer and provide a linking site with the hydroxyethyl acrylate at one end of the silicone oligomer and with the mercaptopropyl silane at the other end.

A preferred application for the composite oligomer is as an oligomeric additive, or even as a main oligomeric component, in a radiation-curable coating, and in particular, an inner primary, optical glass fiber coating. The amount of oligomeric additive incorporated into the radiation curable matrix is not particularly limited but will be sufficient or effective to achieve the specific performance objectives of the particular application. In general, however, a suitable amount will be between about 0.5 wt. % and about 90 wt. %, preferably, between about 0.5 wt. % and about 60 wt. %, and more preferably, between about 0.5 wt. % and about 30 wt. % with respect to the total weight of the radiation-curable coating formulation. In general, higher molecular weight composite oligomers will be present in a radiation-curable coating in greater weight percentages than lower molecular weight composite oligomers.

The composite oligomer functions to tailor the properties of formulations which exhibit too great a coefficient of friction or too low adhesion. Specifically, the composite oligomer can increase the adhesion if the adhesion is unacceptably low, and in particular unacceptably low in the presence of moisture. Alternatively, the composite oligomer can reduce the coefficient of friction of a coating. Conventional coupling additives and slip agents cannot perform this dual function.

If desired, although a reduction in the number of additives is desirable, the composite oligomer can be used in conjunction with conventional coupling and slip agents to improve absolute performance or cost-performance. In a preferred embodiment, for example, the composite oligomeric can be used in conjunction with a functional organosilane compound such as, for example, mercaptopropyl silane. For example, a hydroxybutylvinylether adduct with  $OCN-(CH_2)_3Si(OCH_3)_3$  can also be used together with the composite oligomer.

The composite oligomer can be incorporated into a wide variety of radiation-curable formulations. There are no particular limitations provided that the inventive concept is practiced and advantages accrue. One skilled in the art of formulating radiation-curable coatings will easily be able to incorporate the composite oligomer therein to provide the desired properties.

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In optical glass fiber coating applications, for example, other formulation components generally include:

- (i) at least one multi-functional radiation-curable oligomer, which is a different oligomer than the composite oligomer of the present invention, to provide a cross-linked coating;
- (ii) at least one reactive diluent to adjust the viscosity to a level acceptable for application to optical glass fibers, and
- (iii) at least one photoinitiator.

Additives such as antioxidants, and as already noted, coupling and slip agents may also be utilized.

Radiation-curing is generally rapidly effected with use of ultraviolet light, although the present invention is not so limited, and a person of skill in the art can determine the best cure method. Radiation-curing results in polymerization of at least some of the radiation-curable moieties present in the composite oligomer which covalently links the composite oligomer to itself or, more preferably, other radiation-curable components in the formulation. The chemical processes which occur upon mixing and curing formulations are in some cases complex and may not be fully understood. The present invention, however, is not limited by theory and can be readily understood and practiced by persons of skill in the art. The formulations of the present invention, just like the composite oligomer, can be in pre-cured, partially cured, and in cured states. The term component, which defines additives and compounds used to prepare the formulations, generally refers to starting materials before mixing. After mixing, interactions or even reactions between the components may occur.

The composite oligomer can be incorporated into inner primary coating compositions, outer primary coating compositions, ink compositions and matrix forming compositions. The composite oligomer also can be incorporated into so-called single coating systems.

In general, the coating substrate will be an inorganic or glass substrate, although in principle, other substrates such as polymeric substrates may also be effectively used. The substrate preferably has the capacity to couple with the glass coupling moiety of the oligomeric additive. In a preferred application, the coating substrate is an optical glass fiber, and in particular, a freshly drawn, pristine optical glass fiber. Freshly prepared optical glass fiber is known in the art to be responsive to glass coupling agents. Exemplary methods of coating optical fibers are disclosed in, for example, U.S. Pat. Nos. 4,474,830 and 4,913,859, the complete disclosures of which are hereby incorporated by reference.

The present inventions will be further explained by use of the following non-limiting examples.

#### Example 2-1 and Comparative Examples B-1 and B-2

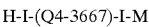
##### Synthesis of Novel Composite Oligomer

A 1,000 mL four-necked flask was charged with isophorone diisocyanate (55.58 g), 2,6-di-tertbutyl-4-methylphenol (0.12 g) and dibutyltin dilaurate (0.24 g) were added to the flask. 14.51 grams of Hydroxyethyl acrylate was added over a 90 minute period while maintaining the temperature below 40 C. At the end of 90 minutes, the temperature was increased to 40 C., and the mixture was stirred at 40 C. for one hour. The temperature was allowed to decrease to about 30 C. Mercaptopropyl silane (28.13 g of an 87.1% pure product) was added over 90 minutes during which time the temperature was maintained below 40 C. After the addition of mercaptopropyl silane, the temperature was increased to 40 C., and the reaction mixture was stirred at 40 C. for 17–18

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hours. 300 g of a 50% ethoxylated polydimethylsiloxane diol of 1200 equivalent weight Q4-3667 (Dow Corning) was then added, and the temperature was increased to 70 C. After about six hours, the isocyanate content was measured to be about zero percent. The temperature was decreased to 50 C. Based on the reaction conditions and reactants, a composite silicone silane acrylate oligomer was formed having the following structure:



wherein:

- H=hydroxyethylacrylate,
- I=isophorone diisocyanate,
- Q4-3667=the above described silicone diol, and
- M=mercaptopropyl silane

Preparation of Pre-Cured Formulation

The components shown in Table 2 were combined, except for the composite oligomer and the silane coupling agent. The components were heated to about 60 C. and mixed to form homogeneous mixtures. The composite oligomer and silicone coupling agent were mixed therein and the mixture was heated for approximately 15 minutes at 60 C. to form an improved radiation-curable, inner primary, optical glass fiber coating composition, Example 2-1. The mixtures for Comparative Examples B-1 and B-2 were prepared similarly. Drawdowns of the compositions were made and then suitably cured by exposure to UV light to form cured coatings. The cured coatings were tested for resistance to delamination and fiber pull-out residue using the following methods.

Water Soak Delamination Test

A drawdown of each inner primary coating composition was made to form a 75 micron film of the inner primary coating composition on microscope slides and then cured by exposure to 1.0 J/sq cm, from a Fusion D lamp, 120 W/cm, under a nitrogen atmosphere. Then, a drawdown of each outer primary coating was made to form a 75 micron film of the outer primary coating composition over the cured 75 micron inner primary film, and then cured in the same manner as the inner primary coating.

Deionized water was placed in a 500 ml beaker and the coated microscope slides were soaked in the water. The beaker containing the coated slides was then placed in a 60 C. hot water bath. The films were observed for delamination periodically. The time when the first signs of delamination appeared were recorded.

Fiber Pull-Out Residue Test

The operation of stripping coatings from optical fibers to leave a bare glass surface was simulated by pulling four bare glass fibers out of a layer of cured inner primary coating. Microscopic examination of the pulled-out fibers at low magnification (e.g., 10x) clearly revealed the presence or absence of debris on the glass surface. If debris was present, the amount of debris was noted. The results of these tests are provided in Table 2.

TABLE 2

Component (amount is % by weight based on total weight of composition)	Ex.2-1	Comp. Ex B-1	Comp. Ex B-2
Urethane acrylate oligomer	53.2	56	53.87
Isodecyl Acrylate	13.3	14	13.47
Ethoxylated-nonylphenol Monoacrylate	24.22	25.5	24.53

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TABLE 2-continued

Component (amount is % by weight based on total weight of composition)	Ex.2-1	Comp. Ex B-1	Comp. Ex B-2
Silicone Silane Oligomer	5	0	0
H-I-Q4-3667-I-A189			
Q4-3667 (Dow Corning)	0	0	3.8
Photoinitiator	2.85	3	2.89
Antioxidant	0.47	0.5	0.48
y-Mercapto-propyl Trimethoxy-Silane	0.95	1.0	0.96
Fiber	no	lot of	no
Pull-out Residue Test	residue	residue	residue
Delamination, if any, after the hot water soak*	none	none	delam. After 1 hour at 60° C.

The oligomers were formed by reacting the following monomers:

- H = Hydroxyethyl Acrylate
- I = Isophorone Diisocyanate
- Q4-3667 = ethoxylated polydimethylsiloxane diol, Mw of 1200 (Dow Corning)

\*Samples were aged for 4 hours at 60° C. Then the water bath was shut-off for about 70 hours. The temperature was then brought back to 60° C. for an additional 48 hours.

Comparative Example B-1 was a formulation which did not contain the composite oligomer of the present invention, but which contained a silane coupling agent. However, poor results were obtained in the pull-out test because adhesion was too strong.

Comparative Example B-2 was a formulation which contained a conventional silicone slip agent. The silicone slip agent improved the results of the pull-out test compared to Comparative Example A, but only at the expense of hydrolytic interfacial adhesion.

Example 2-1 was a formulation that contained the composite oligomer of the present invention. The composite oligomer remarkably improved the results of the pull-out test but not at the expense of hydrolytic interfacial adhesion.

Examples 2-2 and 2-3, and Comparative Examples B-3 and B-4

These Examples and Comparative Examples were conducted to demonstrate the effect of the composite oligomer on glass plate adhesion. The formulations shown in Table 3 were prepared in the same manner as in Example 2-1 and Comparative Examples B-1 and B-2. The silicone silane acrylate oligomer was prepared in the same manner as in Example 2-1, except that a silicone diol HSI-2111 (Tego Chemie) was used instead of Q4-3667 (Dow Corning).

Films of the coating materials (75 microns thick) were prepared on microscope slides and then cured by exposure to UV light. A commercially available outer primary coating was formed on top of the coatings. The films were soaked in water at 60 C. and then examined for delamination. In addition, dry and wet adhesion was measured at 50% and 95% relative humidity (RH), respectively. The results are summarized in Table 3.

Dry (50% RH) and wet (95% RH) adhesion can be measured by recognized test methods. For example, as explained in U.S. Pat. No. 5,336,563 (Coady et al.) and U.S. Pat. No. 5,384,342 (Szum), the wet and dry adhesion was tested on cured film samples prepared by drawing down, with a Bird Bar, a 75 micron film of the coating compositions on glass microscope slides and cured by exposure to 1.0 J/sq cm, from a Fusion D lamp, 120 W/cm, under a nitrogen atmosphere, as noted above in Example 2-1.

The samples were then conditioned at a temperature of 23±2° C. and a relative humidity of 50±5% for a time period

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of 7 days. A portion of the film was utilized to test dry adhesion. Subsequent to dry adhesion testing, the remainder of the film to be tested for wet adhesion was further conditioned at a temperature of 23±2° C. and a relative humidity of 95% for a time period of 24 hours. A layer of polyethylene wax/water slurry was applied to the surface of the further conditioned film to retain moisture.

The adhesion test was performed utilizing apparatus which included a universal testing instrument, e.g., an Instron Model 4201 commercially available from Instron Corp., Canton, Mass., and a device, including a horizontal support and a pulley, positioned in the testing instrument.

After conditioning, the samples that appeared to be uniform and free of defects were cut in the direction of the draw down. Each sample was 6 inches long and 1 inch wide and free of tears or nicks. The first one inch of each sample was peeled back from the glass. The glass was secured to the horizontal support with the affixed end of the specimen adjacent the pulley. A wire was attached to the peeled-back end of the sample, run along the specimen and then run through the pulley in a direction perpendicular to the specimen. The free end of the wire was clamped in the upper jaw of the testing instrument which was then activated. The test was continued until the average force value, in grams force/inch, became relatively constant. The preferred value for wet adhesion is at least about 5 g/in.

TABLE 3

Component (Amount in parts by weight)	Ex.2-2	Ex.2-3	Comp. Ex. B-3	Comp. Ex. B-4
Oligomer C	49.22	49.22	49.22	49.22
H-I(PTHF2000-I) <sub>2</sub> -H				
Ethoxylated nonylphenol Acrylate	24.76	24.76	24.76	24.76
Lauryl Acrylate	16.64	16.64	16.64	16.64
2,4,6- trimethylbenzoylD iphenyl Phosphine Oxide	3.0	3.0	3.0	3.0
Thiodiethylene bis (3,5-di-Tert- Butyl-4- Hydroxy)hydrocinn amate	0.46	0.46	0.46	0.46
gamma- Mercaptopropyl Trimethoxy Silane	0.92	—	0.92	
Silicone Silane	5	5	—	—
Acrylate Oligomer				
H-I-HSi2111-I-M				
Adhesion at 50% RH (g/in)	45	14	27	9
Adhesion at 95% RH (g/in)	34	12	20	4
60° C. Water Soak	No Delamin ation After 24 Hours	Slight Delamin ation After 15 Minutes	No Delaminati on After 8 Hours; Slight Delaminati on After 24 Hours	Delaminat ion After 15 Minutes

The oligomers were formed by reacting the following monomers:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
M = Mercapto Silane  
PTHF2000 = 2000 molecular weight Polytetramethylene Ether Glycol (BASF)  
HSi2111 = a silicone diol having a MW of 1000 (Tego Chemie)

The results in Table 3 indicate that the composite oligomer is not only able to improve adhesion to the glass surface,

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but is also able to act synergistically with a conventional silane coupling agent.

Example 2-4

The formulation shown in Table 4 was prepared in the same manner as in Example 2-1. The silicone silane acrylate oligomer was the same as that prepared in Example 2-1.

A film of the coating material (75 micron thick) was prepared on glass plates and then cured by exposure to UV light in the same manner as above. The tensile strength, elongation and modulus were measured.

A 75 micron film of the coating material was also prepared and suitably cured. The crack propagation was then measured. A fiber pull-out friction test was also conducted, as described herein. The predicted ribbon strip cleanliness was calculated. The results are shown in Table 4.

TABLE 4

Component (Amount in % by weight of total composition)	Example 2-4
H-I-(PTGL2000-I) <sub>2</sub> -H	49.24
Ethoxylated Nonylphenol Acrylate Ester	25.46
Diphenyl (2,4,6-trimethylbenzoyl) Diphenyl	3
Phosphine Oxide and 2-Hydroxy-2-Methyl-1- Phenyl-1-Propanone blend	
Lauryl Acrylate	16
Thiodiethylene bis (3,5-di-Tert-Butyl-4- Hydroxy)hydrocinnamate	0.5
H-I-HSi2111-I-M	5
Mercaptopropyl trimethoxy silane	0.8
Test Results	
Viscosity, mPa.s at 25° C.	7000
Tensile Strength, MPa	0.8
Elongation, %	230
Modulus, MPa	1
Dose@95% Modulus, J/cm <sup>2</sup>	0.64
E' = 1000 MPa, ° C.	-66
E' = 100 MPa, ° C.	-50
Peak TAN Delta ° C.	-40
E <sub>0</sub> , MPa	1.3
Strip Cleanliness Predicted	3
Crack Propagation, mm	1.49
Fiber Pull-out Friction, g/mm	18.5

The oligomers were formed by reacting the following components:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
M = Mercaptopropyl trimethoxy silane  
PTGL2000 = 2000 molecular weight  
polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl  
copolymer diol (Mitsui, NY)  
HSi2111 = a silicone diol having a Mw of 1000 (Tego Chemie, or Gold Schmidt Chemical Corp.)

From the above test data, surprisingly, the novel silicon silane acrylate oligomer can be used to provide an inner primary coating having a fiber friction that provides a resistive force that is less than the cohesive strength of the inner primary coating. This finding is based on the predicted strip cleanliness being about 3. A value of about 3 or less is very good and will usually provide a bare optical glass fiber which is suitable for connection to another optical glass fiber or component of a light transmission assembly, without having to wipe residue from the bare optical glass fiber.

DESCRIPTION OF TEST METHODS USED  
HEREIN

Predicted Strip Cleanliness Test Method

The predicted strip cleanliness is the predicted degree of cleanliness of a bare optical glass fiber after the inner primary coating has been removed during ribbon stripping. A lower number is better.

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Surprisingly, it has been found that the degree of cleanliness of a bare optical glass fiber of a selected ribbon assembly can be predicted by measuring the following two properties of the inner primary coating:

- (1) fiber pull-out friction; and
- (2) crack propagation.

The crack propagation is a measure of the cohesive strength of the inner primary coating. The greater the cohesive strength of the inner primary coating the greater the amount of energy required to break apart the inner primary coating. Thus, an inner primary coating having a higher cohesive strength can withstand greater stripping forces during ribbon stripping without breaking apart and leaving residue on the surface of the optical glass fiber, than an inner primary coating having a lower cohesive strength.

The crack propagation can be measured as follows. First make a 75 micron thick drawdown of the inner primary composition and then cure the film by exposing it to 1.0 J/cm<sup>2</sup> of UV from a Fusion D lamp under a nitrogen atmosphere. Cut three test strips of dimensions 35 mm long, 12 mm wide, and 75 micron thick. A cut 2.5 mm long is made in the side of each strip. A strip is mounted in a RSA-II rheometer, the temperature brought to 90 C. (representative ribbon stripping temperature), and a constant extension rate of 0.1 mm/second is applied to the test strip. The measure of cohesive strength is the increase in length L before the crack propagates across the width of the test strip. The gauge length is constant at 23.2 mm. The value reported is currently the average of three measurements.

The fiber pull-out friction of the inner primary coating is an estimate of the fiber friction between the inner primary coating and the bare optical glass fiber. In general, the lower the fiber pull-out friction of the inner primary coating the lower the fiber friction between the optical glass fiber and the inner primary coating, the lower the resistive force, and the easier the inner primary coating will slide off of the optical glass fiber. Also, the lower the fiber friction, the less force that will be applied to the inner primary coating to conduct ribbon stripping. The less the force being applied to the inner primary coating, the lower the chance that the cohesiveness of the inner primary coating will fail, thus leaving inner primary coating residue on the surface of the optical glass fiber.

The fiber pull-out friction test can be performed as follows. The sample consists of a bare, clean optical fiber, one end of which has been embedded in a 250 micron thick sheet of cured inner primary coating to be tested. This assembly is mounted in a suitable instrument such as a Rheometrics RSA-II rheometer, and the temperature raised to a representative ribbon stripping temperature (such as 90° C.), and the fiber pulled slowly out of the sheet at a rate of 0.1 mm/sec. The instrument records and plots force vs distance. The plots typically show a linear region of negative slope, which is the result of a decreasing area of contact between fiber and coating, as the fiber is being withdrawn. The slope is measured, and is the output of the test. Low slope values correspond to a low fiber pull-out friction, and vice versa. Three test samples should be performed and their average used as the final output of the test.

Prior to using the information from the crack propagation and coefficient of friction measurements as a prediction method, calibration is required. Calibration consists of obtaining test data on at least five inner primary coatings of known cleanliness performance, and fitting the data to a three-dimensional surface using statistical procedures in a suitable statistical/plotting computer program. A convenient two-dimensional representation of the three-dimensional

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surface is a contour plot, in which each contour represents a fixed value of the cleanliness rating, and the vertical and horizontal axes are output values of the fiber pull-out friction and crack propagation tests, respectively.

The cleanliness ratings should be expressed on a quantitative scale, for example, a scale of 1 to 5. An example of a suitable quantitative scale is the "Mill's" test described in the background section herein above, the complete disclosure of which is incorporated herein by reference. When referring to strip cleanliness and predicted strip cleanliness herein, the numerical values correspond to those of the Mill's test.

After the calibration contour plot has been obtained, a point is plotted on it, using data from crack propagation and fiber pull-out friction measurements of an inner primary coating formulation for which a cleanliness prediction is desired. A prediction of cleanliness is obtained by noting the position of the point relative to the contour lines closest to it.

The following hypothetical example illustrates the calibration step, and the use of the contour plot thus produced, to obtain a predicted ribbon-stripping cleanliness. Eight inner primary coatings A through H, are prepared, and coated on an optical fiber in the usual manner, all having the same outer primary coating over the respective inner primary coatings. Coated fiber representing each of the eight inner primary coatings are then coated with an ink layer, and then assembled into a ribbon assembly. The type of ink and matrix material for ribbon assembly should be identical for all eight specimens. Three ribbon assemblies of each sample can be stripped at the desired ribbon stripping temperature. The cleanliness of each sample is evaluated using the Mill's test, in which 1 is best and 5 is worst. The final rating for each of the eight specimens is the average of the ratings of the three replicates. The hypothetical results are shown in Table 5.

TABLE 5

(HYPOTHETICAL)			
Coating	Fiber Friction (g/mm)	Crack Propagation (mm)	Rating
A	2	1.1	1.6
B	30	0.8	5
C	35	1.6	3.7
D	20	1.7	2.8
E	7	2.3	1.8
F	25	2.0	1.5
G	4	1.6	1.6
H	22	1.0	3.9

Next, samples for fiber pull-out friction and crack propagation are prepared from the inner primary coating made from the selected inner primary composition, and output values for each test obtained, by the methods described herein. At this point, there are three data values associated with each of the eight samples. Hypothetical values, chosen to be typical of actual cases, are recorded in Table 5. The contour plot produced by a statistical software program is shown in FIG. 6.

This contour plot is used as follows. For example, a sample of an experimental inner primary coating is measured by the Fiber Pull-Out Friction and Crack Propagation tests, and the resulting data values were 10 and 1 respectively. The point corresponding to those values is located on the contour plot, and it is seen to fall between the contour values of 2.5 and 3. From its location relative to the two lines, the predicted cleanliness rating is estimated to be about 2.7.

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A value of about 3 or less is considered acceptable for optical glass fiber connections.

#### Viscosity Test Method

The viscosity was measured using a Physica MC10 Viscometer. The test samples were examined and if an excessive amount of bubbles was present, steps were taken to remove most of the bubbles. Not all bubbles need to be removed at this stage, because the act of sample loading introduces some bubbles.

The instrument was set up for the conventional Z3 system, which was used. The samples were loaded into a disposable aluminum cup by using the syringe to measure out 17 cc. The sample in the cup was examined and if it contains an excessive amount of bubbles, they were removed by a direct means such as centrifugation, or enough time was allowed to elapse to let the bubbles escape from the bulk of the liquid. Bubbles at the top surface of the liquid are acceptable.

The bob was gently lowered into the liquid in the measuring cup, and the cup and bob were installed in the instrument. The sample temperature was allowed to equilibrate with the temperature of the circulating liquid by waiting five minutes. Then, the rotational speed was set to a desired value which will produce the desired shear rate. The desired value of the shear rate is easily determined by one of ordinary skill in the art from an expected viscosity range of the sample.

The instrument panel read out a viscosity value, and if the viscosity value varied only slightly (less than 2% relative variation) for 15 seconds, the measurement was complete. If not, it is possible that the temperature had not yet reached an equilibrium value, or that the material was changing due to shearing. If the latter case, further testing at different shear rates will be needed to define the sample's viscous properties. The results reported are the average viscosity values of three test samples.

#### Tensile Strength, Elongation and Modulus Test Method

The tensile strength, elongation and modulus of cured samples was tested using a universal testing instrument, Instron Model 4201 equipped with a personal computer and software "Series IX Materials Testing System." The load cells used were 2 and 20 pound capacity. The ASTM D638M was followed, with the following modifications.

A drawdown of each material to be tested was made on a glass plate and cured using a UV processor. The cured film was conditioned at 22 to 24° C. and 50±5% relative humidity for a minimum of sixteen hours prior to testing.

A minimum of eight test specimens, having a width of 0.5±0.002 inches and a length of 5 inches, were cut from the cured film. To minimize the effects of minor sample defects, sample specimens were cut parallel to the direction in which the drawdown of the cured film was prepared. If the cured film was tacky to the touch, a small amount of talc was applied to the film surface using a cotton tipped applicator.

The test specimens were then removed from the substrate. Caution was exercised so that the test specimens were not stretched past their elastic limit during the removal from the substrate. If any noticeable change in sample length had taken place during removal from the substrate, the test specimen was discarded.

If the top surface of the film was talc coated to eliminate tackiness, then a small amount of talc was applied to the bottom surface of test specimen after removal from the substrate.

The average film thickness of the test specimens was determined. At least five measurements of film thickness were made in the area to be tested (from top to bottom) and

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the average value used for calculations. If any of the measured values of film thickness deviates from the average by more than 10% relative, the test specimen was discarded. All specimens came from the same plate.

The appropriate load cell was determined by using the following equation:

$$[A \times 145] \times 0.0015 = C$$

Where:

A=Product's maximum expected tensile strength (MPa);  
145=Conversion Factor from MPa to psi;

0.00015=approximate cross-sectional area (in<sup>2</sup>) of test specimens; and

C=lbs.

The 2 pound load cell was used for materials where C=1.8 lbs. The 20 pound load cell was used for materials where 1.8<C<18 lbs. If C>19, a higher capacity load cell was required.

The crosshead speed was set to 1.00 inch/min (25.4 mm/min), and the crosshead action was set to "return at break". The crosshead was adjusted to 2.00 inches (50.8 mm) jaw separation. The air pressure for the pneumatic grips was turned on and adjusted as follows: set approximately 20 psi(1.5 Kg/cm<sup>2</sup>) for primary optical fiber coatings and other very soft coatings; set approximately 40 psi(3 Kg/cm<sup>2</sup>) for optical fiber single coats; and set approximately 60 psi(4.5 Kg/cm<sup>2</sup>) for secondary optical fiber coatings and other hard coatings. The appropriate Instron computer method was loaded for the coating to be analyzed.

After the Instron test instrument had been allowed to warm-up for fifteen minutes, it was calibrated and balanced following the manufacturer's operating procedures.

The temperature near the Instron Instrument was measured and the humidity was measured at the location of the humidity gage. This was done just before beginning measurement of the first test specimen.

Specimens were only analyzed if the temperature was within the range 23±1.0 C. and the relative humidity was within 50±5%. The temperature was verified as being within this range for each test specimen. The humidity value was verified only at the beginning and the end of testing a set of specimens from one plate.

Each test specimen was tested by suspending it into the space between the upper pneumatic grips such that the test specimen was centered laterally and hanging vertically. Only the upper grip was locked. The lower end of the test specimen was pulled gently so that it has no slack or buckling, and it was centered laterally in the space between the open lower grips. While holding the specimen in this position, the lower grip was locked.

The sample number was entered and sample dimensions into the data system, following the instructions provided by the software package.

The temperature and humidity were measured after the last test specimen from the current drawdown was tested. The calculation of tensile properties was performed automatically by the software package.

The values for tensile strength, % elongation, and (secant or segment) modulus were checked to determine whether any one of them deviated from the average enough to be an "outlier." If the modulus value was an outlier, it was discarded. If there were less than six data values for the tensile strength, then the entire data set was discarded and repeated using a new plate.

#### Elastic Modulus Test Method

The elastic modulus (E'), the viscous modulus (E''), and the tan delta (E''/E'), which is an indication of the material's

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T<sub>g</sub> of the examples were measured using a Rheometrics Solids Analyzer (RSA-11), equipped with: 1) A personal computer having MS-DOS 5.0 operating system and having Rhios® software (Version 4.2.2 or later) loaded; 2) A liquid nitrogen controller system for low-temperature operation.

The test samples were prepared by casting a film of the material, having a thickness in the range of 0.02 mm to 0.4 mm, on a glass plate. The sample film was cured using a UV processor. A specimen approximately 35 mm (1.4 inches) long and approximately 12 mm wide was cut from a defect-free region of the cured film. For soft films, which tend to have sticky surfaces, a cotton-tipped applicator was used to coat the cut specimen with talc powder.

The film thickness of the specimen was measured at five or more locations along the length. The average film thickness was calculated to  $\pm 0.001$  mm. The thickness cannot vary by more than 0.01 mm over this length. Another specimen was taken if this condition was not met. The width of the specimen was measured at two or more locations and the average value calculated to  $\pm 0.1$  mm.

The geometry of the sample was entered into the instrument. The length field was set at a value of 23.2 mm and the measured values of width and thickness of the sample specimen were entered into the appropriate fields.

Before conducting the temperature sweep, moisture was removed from the test samples by subjecting the test samples to a temperature of 80 C. in a nitrogen atmosphere for 5 minutes. The temperature sweep used included cooling the test samples to about -60 C. or about -80 C. and increasing the temperature at about 1/minute until the temperature reached about 60 C. to about 70 C. The test frequency used was 1.0 radian/second.

## Soluble Wax

Wax can be added as a slip agent to adjust the fiber friction between the inner primary coating and the surface of the optical glass fiber to a value that results in a resistive force that is less than the cohesive strength of the inner primary coating. However, conventional waxes exhibit incompatibility problems with inner primary coatings. Many waxes do not dissolve well in inner primary coatings and therefore they tend to separate out from solution. Furthermore, conventional waxes tend to cause the resulting inner primary coating to be hazy in appearance, which is undesirable. The term "soluble wax" is used herein to designate those waxes which are sufficiently soluble in the inner primary coating composition at the concentration required to provide the desired level of fiber friction. The term "wax" is understood to include waxes as defined in Hawley's "Condensed Chemical Dictionary", 11th edition, the said definition being incorporated herein by reference.

It has been found that by selecting modified waxes or by modifying the waxes, the incompatibility problems can be substantially avoided. In selecting a modified wax, the solubility of the modified wax in the desired inner primary composition should first be considered. Usually, waxes tend to be insoluble in inner primary coating compositions. The solubility of the wax in the inner primary coating will depend mainly upon the following:

- (1) the relative polarity of the wax and the polarity of the monomers and oligomers present in the inner primary composition,
- (2) the respective types of functional groups present in the wax and the monomers and oligomers present in the inner primary composition, and
- (3) the similarity between the molecular structure of the wax and the oligomers or monomers present in the inner primary composition, such as aliphatic/aromatic, unsaturated/saturated, linear/branched, etc., entities.

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For example, the solubility of the wax can be increased by incorporating functional groups which are similar to those present in the oligomers or monomers present in the inner primary composition. If the inner primary composition contains monomers or oligomers having ester groups, then ester groups can be incorporated into the molecular backbone structure of the wax or the ester groups can be grafted onto the backbone of the wax. Alternatively, wax-like, long-chain fatty esters can be used. Commercial examples of suitable fatty esters include:

- Laneto-50 and 100 (PEG-75 lanolin),
- Laneto-AWS (PPG-12-PEG-50 lanolin),
- Ritacetyl (acetylated lanolin),
- Ritahydrox (hydroxylated lanolin),
- Ritasol (isopropyl lanolate),
- Ritalan (lanolin oil),
- Ritalan AWS (PPG-12-PEG-65-lanolin oil),
- Ritawax (lanolin alcohol),
- Supersat (hydrogenated lanolin),
- Forlan C-24 (choleth-24 and Ceteth-24),
- Ritachol 1000 (cetearyl alcohol, polysorbate 60, PEG-150-stearate, and steareth-20),
- Ritapro 100 (cetearyl alcohol, steareth-20, and steareth-10),
- Pationic ISL (sodium isostearoyl lactylate),
- Pationic CSL (calcium stearoyl lactylate),
- Pationic SSL (sodium stearoyl lactylate),
- Pationic SBL (sodium behenoyl lactylate),
- Pationic 138C (sodium lauroyl lactylate),
- Pationic 122A (sodium caproyl lactylate),
- Pationic SCL (sodium cocoyl lactylate),
- Ritox 36 (laureth-23),
- Ritox 52 (PEG-40 stearate),
- Rita CA (cetyl alcohol),
- Rita SA (stearyl alcohol), and
- Rita Cetearyl Alcohol 70/30, (RITA Corp.).

Preferably, the fatty ester modified wax is isocetyl stearate.

If the inner primary composition contains monomers or oligomers having alkoxy or hydroxy groups, then to increase the solubility of the wax, alkoxy or hydroxy groups can be incorporated into the molecular backbone structure of the wax or the alkoxy groups can be grafted onto the backbone of the wax. Commercial examples of such modified waxes include the Unilin™ series of alcohol modified waxes from Petrolite, and

- Ritawax (lanolin alcohol),
- Ritachol 1000 (cetearyl alcohol, polysorbate 60, PEG-150-stearate, and steareth-20),
- Ritapro 100 (cetearyl alcohol, steareth-20, and steareth-10),
- Rita CA (cetyl alcohol),
- Rita SA (stearyl alcohol), and
- Rita Cetearyl Alcohol 70/30, (RITA Corp.).

Preferably, the alkoxy modified wax is polypropyleneglycol<sub>12</sub>polyethyleneglycol<sub>50</sub>lanolin.

- As another example, if the inner primary composition contains monomers or oligomers having amine groups, then to increase the solubility of the wax, amine groups can be incorporated into the molecular backbone structure of the wax or the amine groups can be grafted onto the backbone of the wax. An example of such a modified wax is the Armeen® series of amine modified waxes (Armak), such as
- Armeen TD (tallowamine),
- Armeen O, OL or OD (oleylamines),
- Armeen SD (soyaamine),
- Armeen 18 (octadecylamine),
- Armeen HT, HTD or 2HT (hydrogenated tallow),
- Armeen T or TM-97 (tallowamine),
- Armeen 12D (dodecylamine),



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Armeen C or CD (cocoamine),  
Armeen 16D (hexadecylamine),  
Armeen 2C (dicocoamine),  
Armeen M2C (methylcococoamine),  
Armeen DM12D (dimethyldodecylamine),  
Armeen DMCD or DMMCD (dimethylcococoamine),  
Armeen DM14D (dimethyltetradecylamine),  
Armeen DM16D (dimethylhexadecylamine),  
Armeen DM18D (dimethyloctadecylamine),  
Armeen DMHTD (dimethyl(hydrogenatedtallow)amine),  
Armeen DMTD (dimethyltallow amine),  
Armeen DMSD (dimethylsoyamine) or  
Armeen DMOD (dimethyltallow amine).  
Preferably, the amine substituted wax is methyl  
di(hydrogenated tallow)amine.

An example of a further functional group that can be  
incorporated into the wax includes carboxylic acids. Suit-  
able examples of saturated modified waxes include capric  
acid, lauric acid, myristic acid, palmitic acid, and stearic  
acid. Examples of suitable unsaturated waxes include oleic  
acid, ricinoleic acid, linoleic acid, and linolenic acid.

The functional groups present on the modified wax do not  
necessarily have to be identical with those present in the  
oligomers or monomers of the inner primary coating com-  
position in order to achieve increased solubility. Functional  
groups having similar properties, such as hydrogen bonding,  
polarity, etc., can be mixed and matched as desired to  
increase solubility.

The solubility of the wax can also be increased by  
modifying a wax or selecting a wax having a similar  
molecular structure to that of the monomers and oligomers  
present in the inner primary composition. For example, if the  
monomers and oligomers contain aromatic groups, the wax  
can be selected or modified to contain aromatic groups. If  
the monomers or oligomers contain substantial amounts of  
unsaturation, then the wax can be modified or selected to  
contain substantial amounts of unsaturation. Furthermore, if  
the monomers or oligomers are substantially linear, then a  
substantially linear wax can be utilized. Commercial examples  
of substantially linear waxes include Polymekon, Ceramer  
67 and 1608, and Petrolite C-400, CA-11, WB-5, WB-11,  
and WB-17 (Petrolite).

Based on the teachings provided herein, one skilled in the  
art will be able to modify or select the desired wax, and to  
use the selected wax in an amount to provide the desired  
level of fiber friction between the inner primary coating and  
the surface of the optical glass fiber. The amount of the wax  
present in the inner primary composition will depend on (1)  
the ability of the wax to impart the desired reduction in the  
fiber friction between the inner primary coating and the  
surface of the optical glass fiber, and (2) the solubility of the  
wax in the inner primary composition. The greater the  
solubility of the wax in the inner primary composition, the  
greater the amount of wax that can be present. The greater  
the ability of the wax to reduce fiber friction, the less wax  
that will be required. Preferably, the amount of wax present  
is about the minimum amount necessary to provide a level  
of fiber friction necessary to result in a resistive force that  
provides a clean, residue free optical glass fiber after ribbon  
stripping. As discussed above, the fiber friction level that  
results a resistive force level which will provide a clean,  
optical glass fiber after ribbon stripping depends on the  
cohesive strength of the inner primary coating. The greater  
the cohesive strength of the inner primary coating, the  
greater the amount of resistive force that can be tolerated and  
still provide a clean, bare optical glass fiber after ribbon  
stripping. The amount of wax necessary to provide a fiber

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friction that results in such a level of resistive force can be  
readily determined by one skilled in the art by making test  
samples of ribbon assemblies having different concentra-  
tions of the selected wax in the inner primary coating. The  
amount of wax required should be determined using com-  
plete ribbon structures because, as discussed hereinabove,  
the presence of the outer primary coating will have an effect  
on the strippability of the inner primary coating.

Suitable amounts of wax can also be closely approxi-  
mated by using the fiber pull-out friction and crack propa-  
gation test methods described herein, in which the amounts  
of wax that provide a predicted strip cleanliness of less than  
about 3 are preferred.

It has been found that suitable amounts of modified wax  
include from about 0.01% to about 10% by weight of the  
total inner primary composition, more preferably about  
0.01% to about 5%, and most preferably about 0.01% to  
about 2%.

If desired, the wax can be further modified to include a  
radiation-curable functional group that can copolymerize  
with radiation-curable monomers and oligomers present in  
the inner primary composition. An example of such a  
radiation-curable functional wax is stearyl acrylate. The  
radiation-curable functional group in general does not have  
to be an acrylate group, but can be any known radiation-  
curable functional group, including those described herein.

The invention will be further explained by the following  
non-limiting examples illustrating the use of waxes.

Examples 3-1 Through 3-4

The components shown in Table 6 were combined to form  
four inner primary coating compositions. Drawdowns of the  
inner primary coating compositions were made and then  
cured by exposure to UV light from a Fusion D lamp, under  
a nitrogen atmosphere. The crack propagation and fiber  
friction for each of the films were tested in the same manner  
as above, and the predicted strip cleanliness was calculated.  
The results are shown in Table 6.

TABLE 6

COMPONENT (Amount in % by weight of total composition)	Example 3-1	Example 3-2	Example 3-3	Example 3-4
Linear Urethane Acrylate	23	—	—	—
Oligomer Having a Weight Average Molecular Weight of 5000, Urethane Acrylate Oligomer H-I-PTGL2000-I- PTGL2000-I-H	—	51.9	42.3	42.3
Lauryl Acrylate	—	16	—	—
Ethoxylated	64.4	25.6	46.2	46.2
Nonylphenol Acrylate	—	—	—	—
Glyceryl Propoxy	8	—	—	—
Triacrylate	—	—	5	5
Phenoxyethyl Acrylate	3	3	3	3
2,4,6-Trimethyl PhenylbenzoylDiphenyl Phosphine Oxide	—	—	—	—
Thiodiethylene bis (3,5-di-tert- butyl-4-hydroxy) hydrocinnamate	.5	0.5	0.5	0.5
Polyethylene/maleic anhydride	.1	—	—	—

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TABLE 6-continued

COMPONENT (Amount in % by weight of total composition)	Example 3-1	Example 3-2	Example 3-3	Example 3-4
copolymer wax (ceramer 1608)				
Methyl di(hydrogenated tallow)	—	2	—	—
Amine				
Isocetyl Stearate	—	—	2	—
PPG <sub>12</sub> PEG <sub>50</sub>	—	—	—	2
Lanolin				
Mercaptopropyl	1	1	1	1
Trimethoxy Silane				
Test results				
Clarity	Clear	Clear	Clear	Clear
Viscosity (mpa.s, 25° C.)		7650	6760	7390
Fiber Friction (g/mm)		7.7	11.4	7.2
Crack Propagation (mm)		1.53	1.56	1.69
Predicted Strip		2.1	2.5	1.9
Cleanliness				
Fiber Pull-out	2.3			
Residue Test				

The oligomers were formed by reacting the following components:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
PTGL2000 = 2000 molecular weight polymethyltetrahydrofurfuryl/  
polytetrahydrofurfuryl copolymer diol (Mitsui, NY), the methyl group pro-  
vides branching which reduces the orientation of the polymers formed  
from the oligomer

The fiber pull-out residue test was the same test as used previously except that the evaluation was quantified on a scale of 0 to 10, where 0 is the best (no visible residue under 10x magnification) and 10 is the worst (lots of visible residue without use of magnification).

The test results in Table 6 demonstrate that modified waxes can be used to adjust the fiber friction to a level that provides a resistive force less than the cohesive strength of the inner primary coating, which is shown by the excellent predicted strip cleanliness values of less than about 3. Radiation-Curable, Silicone Containing Oligomers and Use of Non-Radiation-Curable Silicone Compounds

Radiation-curable, silicone containing monomers and oligomers can also be used to adjust the level of fiber friction and thereby improve ribbon strippability of the inner primary coating. The radiation-curable, silicone oligomer comprises a silicone compound to which at least one radiation-curable functional group is bound. Preferably, two or more radiation-curable functional groups are connected to the silicone entity.

Preferably the radiation-curable functional group is capable of copolymerizing with the radiation-curable monomers and oligomers present in the inner primary composition when exposed to suitable radiation. Therefore, the selection of the functional group will depend on the monomer or oligomer present in the inner primary composition. One skilled in the art will readily be able to determine which functional groups will cross-link with the monomer or oligomer present in the inner primary composition. While not being limited thereto, examples of suitable functional groups are groups containing vinyl, acrylate, methacrylate, maleate, vinyl ether, or acrylamides, as well as those described herein above.

Examples of commercially available silicone compounds containing a radiation-curable functional group are silicone acrylates Ebecryl 350 and Ebecryl 1360 (Radcure Industries), Tego Rad 2100, 2200, 2500, and 2600 (Tego Chemie), and Coat-O-Sil 3503 (OSI Specialties).

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Alternatively, based on the teachings herein, one skilled in the art will be able to modify known silicone compounds to include the required radiation-curable functionality. For example, a silicone compound provided with hydroxy functionality can be reacted with a diisocyanate compound and a compound containing a hydroxy and a radiation-curable functionality to provide a radiation-curable functionality to said silicone compound. Specific examples include reacting a silicone compound containing a hydroxy functionality with a diisocyanate and hydroxyethylacrylate to provide an acrylate functionality on the silicone compound, or isocyanate and hydroxybutylvinylether to provide a vinyl ether functionality on the silicone compound. Example of suitable silicone compound containing hydroxyl functionality include: polydimethylsiloxane diol of 1200 equivalent weight Q4-3667, DC 193 and DC 1248 (Dow Corning), HSi2111 (Tego Chemie), and Coat-O-Sil 3500 and 3505 (Osi Specialties).

Alternatively, non-radiation-curable silicone compounds (hereinafter referred to as “non-reactive silicone”) can be used to adjust the fiber friction and thereby improve ribbon strippability of the inner primary coating.

U.S. Pat. No. 4,496,210, which is incorporated herein by reference, discloses examples of suitable non-reactive silicones that can be used. Non-reactive silicones can be used separately or in conjunction with the radiation-curable silicone oligomers described herein.

The radiation-curable silicone oligomer and/or non-reactive silicone should be present in an amount to provide a fiber friction that results in a resistive force that is less than the cohesive strength of the inner primary composition. The amount of radiation-curable silicone oligomer and/or non-reactive silicone is preferably the minimum amount required to provide a fiber friction that results in a resistive force less than the cohesive strength of the inner primary composition. Such minimum amount can easily be determined by making test runs of inner primary compositions in which the amount of radiation-curable silicone oligomers and/or non-reactive silicones present is varied. The lowest amount of radiation-curable silicone oligomers and/or non-reactive silicones present which provides a fiber friction that results in a resistive force that is less than the cohesive strength of the inner primary coating is the preferred amount.

A long chain silicone compound containing on average about one radiation-curable functional group (monofunctional) bound near a terminus of the silicone compound can provide further advantages. The end of the long silicone chain furthest from the radiation-curable functional group can be mechanically bound in the inner primary coating. However, upon heating during ribbon stripping, it is believed that the end of the long silicone chain farthest from the radiation-curable functional group can become unbound and diffuse toward the optical glass fiber/inner primary coating interface which is in the direction the heat is propagating. This diffusion of silicone increases at the critical moment during ribbon stripping to facilitate the clean removal of the entire coating system. The silicone acts as a lubricant between the surface of the optical glass fiber and the inner primary coating.

The thickness of an inner primary coating usually varies from about 10 microns to about 35 microns. Thus, a monofunctionalized silicone fluid having a molecular chain length of about 50,000 to about 350,000 Daltons can diffuse toward the glass/inner primary coating interface during ribbon stripping.

Suitable amounts of radiation-curable silicone oligomers and/or non-reactive silicones can also be closely approxi-

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mated by using the friction and crack propagation test methods described herein, in which the amounts of radiation-curable silicone oligomers and/or non-reactive silicones that provide a predicted strip cleanliness of less than about 3 are preferred.

The amount of radiation-curable silicone oligomer and/or non-reactive silicones will also depend on the selection of the inner primary composition, in particular the initial fiber friction of the selected inner primary coating composition. Generally, the higher the initial fiber friction (no slip additive), the greater the amount of radiation-curable silicone oligomer and/or non-reactive silicone that will be required to lower the fiber friction to a level that provides a resistive force lower than the cohesive strength of the inner primary coating.

In general, the radiation-curable silicone oligomers can be used in greater amounts than non-reactive silicones because it is believed that the radiation-curable silicone oligomer will become bound in the inner primary coating during curing, whereas the non-reactive silicone is free to migrate throughout the cured inner primary coating. Alternatively, the radiation-curable silicone oligomer can be the main oligomer used for forming the inner primary coating. It has been found that suitable amounts of radiation-curable silicone oligomer are between about 0.1 to about 90% by weight, preferably about 0.1 to about 60% by weight, and more preferably about 0.1 to about 30% by weight. In general, higher molecular weight radiation-curable silicone oligomers will be present in a radiation-curable coating in greater weight percentages than lower molecular weight composite oligomers.

Suitable amounts of mono-functionalized monomers have been found to be about 0.1 to about 20% by weight, more preferably about 0.1 to about 10% by weight, and most preferably about 0.1 to about 5% by weight.

Suitable amounts of non-reactive silicone are between about 0.01 to about 10% by weight, preferably about 0.01 to about 5% by weight, and more preferably about 0.01 to about 1% by weight.

The invention will be further explained by the following non-limiting examples illustrating the use of silicone entities.

Example 4-1

The components shown in Table 7 were combined to form an inner primary coating composition. A film of the coating material (75 micron thick) was prepared on glass slides and then cured by exposure to UV light in the same manner as above. The tensile strength, elongation and modulus were measured.

A 75 micron film of the coating material was also prepared and suitably cured. The crack propagation was then

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measured. A friction test was also conducted, as described herein. The predicted ribbon strip cleanliness was calculated. The results are shown in Table 7.

TABLE 7

Component (Amount is % by weight of total composition)	Example 4-1
Oligomer H-DesW-PTHF2900-DesW-H	47.5
Ethoxylated Nonylphenol Acrylate	29
Lauryl Acrylate	14.2
2,4,6-Trimethyl Phenylbenzoyl Diphenyl Phosphine Oxide	3
Silicone Oligomer H-I-HSi2111-I-H	5
y-Mercaptopropyltrimethoxy Silane	.8
Thiodiethylene Bis (3,5-di-tert-Butyl-4-Hydroxy)Hydrocinamate	.5
Test Results	
Viscosity, mPa.s (25° C.)	6040
Tensile Strength, Mpa	1
Elongation, %	140
Modulus, Mpa	1.4
Dose at 95%, Modulus, J/Sq CM	.38
Crack Propagation (mm)	1.7
Fiber Pull-Out Friction (g/mm)	17.1
Predicted Strip Cleanliness	2.5-3

The oligomers were formed by reacting the following components:  
 H = Hydroxyethyl Acrylate  
 DesW = bis 4,4-(isocyanatocyclohexyl)methane  
 I = Isophorone Diisocyanate  
 PTHF2900 = 2900 molecular weight Polytetramethylene Ether (BASF)  
 HSi2111 = a silicone diol having a MW of 1000 (Tego Chemie)

Examples 4-2 Through 4-10

The components shown in Table 8 were combined to form 11 different inner primary coating compositions. The viscosity and clarity of the compositions was determined.

Films of the coating materials (75 micron thick) were prepared on microscope slides and then cured by exposure to UV light in the same manner as above. The tensile strength, elongation and modulus were measured.

Additional films of the coating materials were also prepared and suitably cured. The crack propagation was then measured. A friction test was also conducted, as described herein. The predicted ribbon strip cleanliness was calculated. The results are shown in Table 8.

TABLE 8

Component (Amount is % by weight of total composition)	Ex. 4-2	Ex. 4-3	Ex. 4-4	Ex. 4-5	Ex. 4-6	Ex. 4-7	Ex. 4-8	Ex. 4-9	Ex. 4-10
Oligomer H-I-PTHFCD2000-I-PTHFCD2000-I-H	45.67								
Oligomer H-(I-PPG1025)1.06-(-PERM)1.14-I-H			54.86						
Oligomer H-I-PTGL2000-I-H					60.65				
Oligomer H-I-PPG2010-I-PPG2010-I-H		67.5		70					
Oligomer						51.02	49.23		43

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TABLE 8-continued

Component (Amount is % by weight of total composition)	Ex. 4-2	Ex. 4-3	Ex. 4-4	Ex. 4-5	Ex. 4-6	Ex. 4-7	Ex. 4-8	Ex. 4-9	Ex. 4-10
H-I-PTGL2000-I-PTGL2000-I-H Oligomer (H-I) <sub>3</sub> -TPE4542								78	
Ethoxylated Nonylphenol Acrylate Ester	34.48		24.99		32.85	20.14	24.75	16	50.5
Lauryl Acrylate	14.35		13.72			6.92	16.64		
Isodecyl Acrylate									
Phenoxyethyl Acrylate						16.62			
2.5 Mole Propoxylated Nonyl Phenol Acrylate		25.00		23.5					
25:75 weight/weight of Bis(2,6-Dimethoxybenzoyl)(2,4,4-Trimethylpentyl)Phosphine Oxide and 2-Hydroxy-2-Methyl-1-Phenyl Propanone			2.94	3					
2,4,6-trimethylbenzoyl Diphenyl Phosphine Oxide	3				2.5	3	3	1	3
1-Hydroxycyclohexyl Phenyl Ketone		4						2	
Octadecyl 3,5-Bis(1,1-Dimethylethyl)-4-Hydroxybenzenepropanone	.5	.5							
Thiodiethylene bis(3,5-di-Tert-Butyl-4-Hydroxy)hydrocinnamate			.49		.5	.3			.5
Ditridecylthiodipropionate		1		1					
Free Silicone, DC-193 (Dow Corning)		1	2	1				2	2
Free Silicone, DC-190 (Dow Corning)	1								
Teograd 2100 silicone acrylate					2.5	1	5		
L-77 Polyethylene oxide modified Dimethylsiloxane									
1-Propanethiol,3-(Trimethoxysilyl)	1	1	.98	1	1	1	.92	1	1
Clarity When Made	clear	clear	clear	clear				clear	
Clarity After 24 Hours at 4 C.	clear		clear	clear					
Clarity After 24 Hours at -20 C.	clear		clear	clear					
Clarity After 3 Days at 60 C.	clear		clear	clear					
Viscosity (mPa · s, 25 C.)	8700		5600	8000	9520	7170	6240	8200	
Dose @ 95% Modulus (J/sq. cm)	.77	.46	.45	.32	.36	.45		.2	
Tensile Strength (MPa)		.4			1.5	.6	1.1		
Elongation (%)		50			100	140	180		
Modulus (Mpa)		1.2			2.7	1.1	1.3	2.4	
Fiber Friction (g/mm)	3.1	4.9	2.7	4.4	21	18.4	18.5	3	3.4
Fiber Friction (g/mm) After 7 days, 60 C., dose 95% of dose required for complete cure			1	1.4					
Crack Propagation (mm)	2.1	1.49	1	1.4	1.21	1.82	1.47	1.1	1.9
Crack propagation (mm) after 7 days, 60 C., dose 95 of dose required for complete cure			1.1						
Predicted Strip Cleanliness	1.5-2	1.5	2.2	1.5	3.6	3	3	1.5	1.5

The oligomers were formed by reacting the following components:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
PTHFCD2000 = is PolyTHF containing some carbonate linkages  
PPG1025 = is Polypropyleneoxidediol having an average molecular weight of 1000 (Arco)  
PPG2010 = is Polypropyleneoxidediol having an average molecular weight of 2000 (BASF)  
PTGL2000 = 2000 molecular weight polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl copolymer diol (Mitsui, NY)  
TPE4542 = polypropylene glycol ethylene oxide endcapped triol (BASF)  
Perm = Permanol KM10-1733 polycarbonate/polyether copolymer diol

The test results in Table 8 demonstrate that the radiation-curable silicone oligomers and non-reactive silicones can be used to adjust the fiber friction to a level that provides a resistive force less than the cohesive strength of the inner primary coating, which is shown by the predicted strip cleanliness values of about 3 or less.  
Radiation-curable Fluorinated Oligomers and Fluorinated Materials

The fiber friction between the inner primary coating and the surface of the optical glass fiber can also be significantly reduced by incorporating radiation-curable fluorinated oligomers, monomers and/or non-radiation curable fluorinated materials into the inner primary coating composition. The radiation-curable, fluorinated oligomer or monomer comprises a fluorinated compound to which at least one radiation-curable functional group is bound. Preferably, two

or more radiation-curable functional groups are connected to the fluorinated entity.

Preferably the radiation-curable functional group is capable of copolymerizing with the radiation-curable monomers and oligomers present in the inner primary composition when exposed to suitable radiation. Therefore, the selection of the functional group will depend on the monomer or oligomer present in the inner primary composition. One skilled in the art will easily be able to determine which functional groups will cross-link with the monomer or oligomer present in the inner primary composition. While not being limited thereto, examples of suitable radiation-curable functional groups are groups containing vinyl, acrylate, methacrylate, maleate, vinyl ether, or acrylamides, as well as those described herein above.

Examples of commercially available fluorinated compounds containing at least one radiation-curable functional

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group include perfluoro ethyl acrylate (DuPont), 2-(N-Ethylperfluoro Octane Sulfonamido)Ethyl Acrylate (3M), 1H,1H-pentadecafluorooctyl acrylate (Oakwood Research Chemicals), as well as methacrylate or N butyl acrylate versions of these.

Based on the teachings herein, one skilled in the art will be able to modify a fluorinated compound to include the required radiation-curable functionality. For example, a fluorinated compound provided with hydroxy functionality can be reacted with a diisocyanate compound and a compound containing a hydroxy and a radiation-curable functionality to provide a radiation-curable functionality to said fluorinated compound. Specific examples include reacting a fluorinated compound containing a hydroxy functionality with a diisocyanate and hydroxyethylacrylate to provide an acrylate functionality on the fluorinated compound, or isocyanate and hydroxybutylvinylether to provide a vinyl ether functionality on the fluorinated compound. Examples of suitable fluorinated compounds containing hydroxyl functionality include Fluorolink E (Ausimont), 2-methyl-4,4,4-trifluorobutanol, 1H,1H-pentadecafluoro-1-octanol, 1H,1H-pentafluoropropanol-1, and 1H,1H,12H,12H-perfluoro-1,12-dodecanediol (Oakwood Research Chemicals).

Alternatively, non-radiation-curable fluorinated compounds (hereinafter referred to simply as "fluorinated compounds") can be used to adjust the fiber friction and thereby improve ribbon strippability of the inner primary.

The fluorinated compounds can be used separately or in conjunction with the radiation-curable silicone oligomers or monomers described herein.

The radiation-curable fluorinated oligomer or monomer and/or fluorinated compounds should be present in an amount to provide a fiber friction that results in a resistive force that is less than the cohesive strength of the inner primary composition. The amount of radiation-curable fluorinated oligomer and/or fluorinated compound is preferably the minimum amount required to provide a fiber friction that results in a resistive force less than the cohesive strength of the inner primary composition. Such minimum amount can easily be determined by making test runs of inner primary compositions in which the amount of radiation-curable fluorinated oligomers or monomers and/or fluorinated present is varied. The lowest amount of radiation-curable fluorinated oligomers or monomers and/or fluorinated compounds present which provides a fiber friction that results in a resistive force less than the cohesive strength of the inner primary coating is the preferred amount.

Suitable amounts of radiation-curable fluorinated oligomers or monomers and/or fluorinated compounds can also be closely approximated by using the friction and crack propagation test methods described herein, in which the amounts of radiation-curable fluorinated oligomers or monomers and/or fluorinated compounds that provide a predicted strip cleanliness of less than about 3 are preferred.

The amount of radiation-curable fluorinated oligomer or monomers and/or fluorinated compounds will also depend on the selection of the inner primary composition, in particular the initial fiber friction of the selected inner primary coating composition. Generally, the higher the initial fiber friction (no slip additive), the greater the amount of radiation-curable fluorinated oligomer or monomer and/or fluorinated compounds that will be required to lower the fiber friction to a level that provides a resistive force lower than the cohesive strength of the inner primary coating.

In general, the radiation-curable fluorinated oligomers or monomers can be used in greater amounts than non-reactive fluorinated compounds because it is believed that the

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radiation-curable fluorinated oligomers or monomers will become bound in the inner primary coating during curing, whereas the non-reactive fluorinated compounds are free to migrate throughout the cured inner primary coating.

Alternatively, the radiation-curable fluorinated oligomer or monomer can be the main oligomer used for forming the inner primary coating. It has been found that suitable amounts of radiation-curable fluorinated oligomer or monomer are between about 0.1 to about 90% by weight, preferably about 0.1 to about 60% by weight, and more preferably about 0.1 to about 30% by weight. In general, larger molecular weight oligomers can be used in greater amounts than lower molecular weight oligomers or monomers.

Suitable amounts of fluorinated compounds have been found to be between about 0.01 to about 10% by weight, preferably about 0.01 to about 5% by weight, and more preferably about 0.01 to about 1% by weight.

The invention will be further explained by the following non-limiting examples illustrating the use of fluorinated materials.

Examples 5-1 Through 5-3

The components shown in Table 9 were combined to form 5 different inner primary coating compositions. The viscosity and clarity of the compositions were determined.

Films of the coating materials (75 micron thick) were prepared on glass slides and then cured by exposure to UV light in the same manner as above. The tensile strength, elongation and modulus were measured.

Additional films of the coating materials were also prepared and suitably cured. The crack propagation was then measured. A friction test was also conducted, as described herein. The predicted ribbon strip cleanliness was calculated. The results are shown in Table 9.

TABLE 9

Component (% by weight based on total composition)	Ex. 5-1	Ex. 5-2	Ex. 5-3
Oligomer H-(I-PPG1025) <sub>1.06</sub> -(-PERM) <sub>1.14</sub> -I-H	54.32		55.58
Oligomer H-(I-PPG2010) <sub>2</sub> -I-H		67.75	
Ethoxylated Nonylphenol Acrylate Ester	24.74		25.31
Isodecyl Acrylate	13.58		13.9
2.5 Mole Propoxylated Nonyl Phenol Acrylate		25	
25:75 weight/weight of Bis(2,6-Dimethoxybenzoyl) (2,4,4-Trimethylpentyl) Phosphine Oxide and 2-Hydroxy-2-Methyl-1-Phenyl Propanone	2.91		3
1-Hydroxycyclohexyl Phenyl Ketone		4	
Octadecyl 3,5-Bis(1,1-Dimethylethyl)-4-Hydroxybenzenepropanone		0.50	
Thiodiethylene bis(3,5-di-tert-butyl-4-hydroxy)Hydrocinnamate	.48		.5
Ditridecylthiodipropionate		1.00	
Foralkyl EM-6	3.00		
Tridecafluorooctyl Mecaptan (Elf Autochem)			
Fluorosulfonamide (3M)		0.75	.75
mercaptopropyl trimethoxy silane	0.97	1	1
Clarity as made	Clear	Clear	Clear
Clarity after 24 hours at 4° C.			Clear
Clarity after 24 hours at -			Clear

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TABLE 9-continued

Component (% by weight based on total composition)	Ex. 5-1	Ex. 5-2	Ex. 5-3
20° C.			
Clarity after 3 days at 60° C.			Very Few Incom pat's 6200
Viscosity (mPa.s, 25° C.)			
Dose @ 95% Modulus (J/sq.cm)	.77	0.50	.47
Tensile Strength (MPa)		0.50	
Elongation (%)		88	
Modulus (MPa)		1.20	
Fiber Friction (g/mm)	25.5	8.2	10.5
Fiber Friction (g/mm) After 7 days, 60° C., at dose of 95% of dose for complete cure			1.1
Crack Propagation (mm)	1.32	1.54	1.1
Crack Propagation (mm) after 7 days, 60° C., at dose of 95% of dose for complete cure			1
Predicted Strip Cleanliness	3.0	2.0	2.6

Table Notes:  
The oligomers were formed by reacting the following components:  
H = Hydroxyethyl Acrylate; I = Isophorone Diisocyanate  
PPG1025 = is Polypropyleneoxidediol having an average molecular weight of 1000 (Arco)  
PPG2010 = is Polypropylenediol having an average molecular weight of 2000 (BASF)  
PTGL2000 = 2000 molecular weight polymethyltetrahydrofurfuryl/ polytetrahydrofurfuryl copolymer diol (Mitsui, NY)  
Perm = Permalol KM10-1733 polycarbonate/polyether copolymer diol

Solid Lubricants

Surprisingly, it has been found that solid lubricants can be added to the inner primary composition to reduce the fiber friction between the inner primary coating and the surface of the optical glass fiber. The term “solid lubricant” is used herein to mean that the lubricant is substantially insoluble in the inner primary composition and that the particle or flake shape of the solid lubricant is substantially maintained after curing of the inner primary coating composition.

Usually the solid lubricant is non-reactive with the components of inner primary coating composition. Examples of suitable non-reactive solid lubricants are the following, but not limited thereto: solid organic lubricants including organic polysaccharides such as sodium alginate, polyolefins, polyvinyl alcohol, nylon such as Orgasol (Elf Atochem), solid Teflon particles, and hard waxes such as Rad Wax; solid inorganic lubricants including molybdenum disulfide, graphite, silicates such as talc, clays such as kaolin and mica, silica, and boron nitride.

However, if desired, a reactive solid lubricant can be used. Reactive solid lubricants contain a radiation-curable functional group. Preferably, the radiation-curable functional group is capable of copolymerizing with the radiation-curable monomers or oligomers present in the inner primary composition. The radiation-curable functional group can be, for example, any of the radiation-curable functional groups described herein. Specific examples of suitable reactive solid lubricants include zinc acrylate, molybdenum acrylate, aluminum acrylate, barium acrylate, and chromium acrylate.

The particle size is preferably small enough to avoid microbending caused by the solid particles exerting stresses on the surface of the optical glass fiber during use. Furthermore, the particle size is preferably small enough to avoid causing the inner primary coating to be hazy in appearance. Examples of suitable particle sizes have been found to be about 10 microns or less, preferably about 5 microns or less, and most preferably less than about 2 microns.

Alternatively to the particle size, the hardness of the solid lubricant is preferably low enough to avoid microbending

caused by the solid particles exerting stresses on the surface of the optical glass fiber during use. In general, a softer solid lubricant will be less likely to cause such microbending.

Based on the teachings provided herein, one skilled in the art will easily be able to use the selected solid lubricant in an amount to provide the desired level of fiber friction between the inner primary coating and the surface of the optical glass fiber. The amount of the solid lubricant present in the inner primary composition will depend on the ability of the solid lubricant to impart the desired reduction in the fiber friction between the inner primary coating and the surface of the optical glass fiber, and the amount the fiber friction must be reduced to provide a fiber friction level that results in a resistive force less than the cohesive strength of the inner primary coating. In general, the greater the ability of the solid lubricant to reduce fiber friction, the less solid lubricant that will be required. Preferably, the amount of solid lubricant present is about the minimum amount necessary to provide a level of fiber friction necessary to provide a clean, residue free optical glass fiber after ribbon stripping. As discussed above, the fiber friction level that will provide a clean, optical glass fiber after ribbon stripping will depend on the cohesive strength of the inner primary coating. The greater the cohesive strength of the inner primary coating, the greater the amount of fiber friction, and resulting resistive force, that can be tolerated and still provide a clean, bare optical glass fiber after ribbon stripping. The amount of solid lubricant necessary to provide such a level of fiber friction can be easily determined by one skilled in the art by making test samples of ribbon assemblies having different concentrations of the selected solid lubricant in the inner primary coating. The amount of solid lubricant required should be determined using complete ribbon structures because, as discuss herein above, the presence of the outer primary coating will have an effect on the strippability of the inner primary coating.

Suitable amounts of solid lubricant can also be closely approximated by using the fiber pull-out friction and crack propagation test methods described herein, in which the amounts of solid lubricant that provides a predicted strip cleanliness of less than about 3 are preferred.

It has been found that suitable amounts of solid lubricant include from about 0.1% to about 20% by weight of the total inner primary composition, more preferably about 0.1% to about 10%, and most preferably about 0.1% to about 5%.

Preferably, a surfactant is used in combination with the solid lubricant. Examples of a suitable surfactants include: fluorosulfonamide surfactant (3M), 3,6-dimethyl-4-octyne-3,6-diol (Air Products), linear copolymer of vinylpyrrolidone and long chain alpha olefin (International Specialty Products), Solsperse high MW polymeric dispersing agents (Zeneca), and other well-known anionic, cationic and non-ionic surfactants.

The invention will be further explained by the following non-limiting examples.

Examples 6-1 Through 6-3

The components shown in Table 10 were combined to form 8 different inner primary coating compositions. The viscosity and clarity of the compositions was determined.

Films of the coating materials (3 mil) were prepared on microscope slides and then cured by exposure to UV light in the same manner as above. The tensile strength, elongation and modulus were measured.

75 mm films of the coating materials were also prepared and suitably cured. The crack propagation was then measured. A fiber pull-out friction test was also conducted, as

described herein. The predicted ribbon strip cleanliness was calculated. The results are shown in Table 10.

TABLE 10

Component (Amount is % by weight of total composition)	Ex. 6-1	Ex. 6-2	Ex. 6-3
Oligomer H-(I-PTGL2000) <sub>2</sub> -I-H	36.1	42.3	36.1
Ethoxylated Nonylphenol Acrylate	44.4	46.1	43.9
Phenoxyethyl Acrylate	5	5	5
2,4,6-trimethylbenzoyl Diphenyl Phosphine Oxide and 2-Hydroxy-2- Methyl-1-Phenyl-1-Propanone blend	3	3	3
Thioethylene bis(3,5-di-tert- butyl-4-Hydroxy)Hydrocinnamate	.5	.5	.5
y-Mercaptopropyltrimethoxy Silane	1	1	1
Rad Wax 62EB (33% PE wax in epoxy acrylate)	10		
Fluorosulfonamide Surfactant FC- 430 (3M)		.1	.5
Fluoro A (Micronized PTFE)		2	10
Test results			
Clarity (opaque?)	yes	yes	yes
Color	white	white	white
Viscosity, mPa.s at 25°	5440	7960	7520
Film Opacity, 3 mil	opaque	cloudy	cloudy
Fiber Friction (g/mm)	15.2	8.2	6.6
Crack Propagation (mm)		1.96	2.2
Predicted Strip Cleanliness		2.2	2.4

The oligomers were formed by reacting the following components:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
PTGL200 = 2000 molecular weight  
polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl copolymer diol  
(Mitsui, NY)  
Perm = Permalol KM10-1733 polycarbonate/polyether copolymer diol

The test results in Table 10 demonstrate that solid lubricants can be used to reduce the friction to a level that results in a resistive force less than the cohesive strength of the inner primary coating, which is shown by the predicted strip cleanliness values of about 3 or less.

Use of Novel Slip Agents

The above described novel slip agents can be used alone, in combinations of novel slip agents, as novel slip agents with conventional slip agents, and as novel slip agents in combination with the adjusting the normal force as desired to provide the desired level of fiber friction.

Based on the above experimental data, the composition of the inner primary coating can surprisingly be formulated or selected to provide a fiber friction of about 40 (g/mm) or less, preferably about 30 (g/mm) or less, and more preferably about 20 (g/mm), and most preferably about 10 (g/mm) or less at the desired ribbon stripping temperature, in combination with a crack propagation of the inner primary coating which is greater than about 0.7 mm, preferably greater than about 1 mm, more preferably greater than about 1.5 mm, and most preferably greater than about 2 mm, at the desired/design ribbon stripping temperature, such as 90° C. Table 11 and example 11-1 illustrates a practice according to this invention.

TABLE 11

Component (% weight based on total weight of composition)	Example 11-1
Oligomer H-(I-PTGL2000) <sub>2</sub> -I-H	50.3
Isobornyl Acrylate	10
Ethoxylated Nonylphenol Acrylate Ester	15.1

TABLE 11-continued

Component (% weight based on total weight of composition)	Example 11-1
Thiodiethylene bis (3,5-di-tert-butyl-4-Hydroxy) Hydrocinnamate	0.5
Phenoxy Ethyl Acrylate	20
2,4,6-Trimethylbenzoyl Diphenyl Phosphine Oxide	3
Silicone Fluid <sup>1</sup>	0.1
gamma-Mercaptopropyl Trimethoxy Silane	1
Test Results	
Predicted Strip Cleanliness	2.5
Fiber Friction (g/mm)	18.5
Crack Propagation (mm)	2.07

The oligomers and monomers were formulated from the following components:  
H = Hydroxyethylacrylate  
I = Isophoronediiisocyanate  
PTGL2,000 = polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl copolymer diol having the molecular weight (2,000), (Mitsui, NY)  
Silicone Fluid = Byk333 (BYK Chemie) which is polydimethylsiloxane with terminal polyethylene oxide groups

Linear Oligomers

If desired, ribbon strippability can also be improved by increasing the ability of the inner primary coating to transmit force applied during ribbon stripping. In general, the more efficient the inner primary coating is at transmitting the force applied during the ribbon stripping operation, the less stripping force that need be applied to remove the inner primary coating.

It has now also been found that the use of linear oligomers can improve the effectiveness, and consequently the efficiency, of the inner primary coating to transmit the ribbon stripping forces applied during ribbon stripping operations. In general, to the extent that the molecular structure of the oligomer is designed to be more linear, the more densely the oligomers will pack together when forming the inner primary coating. It has been found that as the oligomers become more densely packed, the more efficiently the inner primary coating can transmit the stripping force applied during ribbon stripping.

The ability of a ribbon assembly to strip cleanly during ribbon stripping can be further improved if the polymers bound in the outer primary coating have the ability to orient upon heating.

Examples of linear, radiation-curable oligomers according to the present invention that provide enhanced strippability can be illustrated by the following formula (4):



wherein:

R<sup>1</sup> and R<sup>3</sup> are organic groupings having radiation-curable functional groups as defined herein, and R<sup>2</sup> is an optional organic radical;

L is a linking group, providing a bridging group such as a urethane, thio-urethane, urea or ester grouping, as defined herein, preferably urethane;

R<sup>2</sup> is a substantially linear carbon-containing entity; and n is about 1 to about 40, preferably about 1 to about 20, and most preferably about 1 to about 10, wherein the molecular weight of [R<sup>2</sup>-L]<sub>n</sub> is about 500 to about 20,000, preferably about 1,000 to about 10,000, and most preferably about 1,500 to about 6,000.

When n is 1, [R<sup>2</sup>-L] can contain, for example, a polyolefin, polyether, polycarbonate, or polyester structure having a molecular weight of about 500 to about 20,000. When n is from about 2 to about 5, [R<sup>2</sup>-L] can include a

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polyolefin, polyether, polycarbonate, or polyester having a molecular weight of about 500 to about 10,000. When n is from about 5 to about 30, [R<sup>2</sup>—L] can represent a polyolefin, polyether, polycarbonate, or polyester having a molecular weight of about 500 to about 4,000.

The linear oligomers according to this invention can be used in an amount suitable to provide the desired level of ribbon stripping performance. The desired amount can easily be found and determined by one skilled in the art by testing different amounts of the selected linear oligomer(s) in an inner primary coating, and optionally in an outer primary coating as well, on optical glass fibers encased in a ribbon assembly. It has generally been found that the linear oligomers according to this invention can be used in amounts of about 0.1 to about 90 wt. %, preferably about 5 to about 80 wt. %, more preferably about 5 to about 60 wt. %, based on the total weight of the inner primary or outer primary composition.

Example 7-1 Through 7-2

The components shown in Table 12 were combined to form an inner primary coating composition. The compositions were cured and the fiber pull-out friction of the cured coating was measured, as defined herein. The test results are shown in Table 12.

TABLE 12

Component (Amount is % by weight of total composition)	Ex-ample 7-1	Ex-ample 7-2
Oligomer H-(I-PTHF2000),-I-H	52.26	52.26
Ethoxylated Nonylphenol Acrylate	15.7	15.67
Lauryl Acrylate	15.19	16.19
n-Vinyl Formamide Isobornyl Acrylate	11.8	0
n-Vinyl Formamide Ethylhexyl Acrylate	0	10.8
25:75 weight/weight of Bis(2,6-Dimethoxybenzoyl) (2,4,4-Trimethylpentyl) Phosphine Oxide and 2-Hydroxy-2-Methyl-1-Phenyl Propanone	3.7	3.7
gamma-Mercaptopropyl Trimethoxy Silane	0.92	0.92
Thioethylene Bis(3,5 di-tert-butyl-4-hydroxyl) Hydrocinnamate (antioxidant)	.46	.46
Test Results		
Fiber Pull-Out Residue Test	0.875	1.25

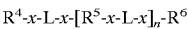
The oligomer was formed by reacting the following components:

H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
PTHF2000 = 2000 molecular weight Polytetramethylene Ether Glycol (BASF)

Terminal Linear Moieties

It has been found that the use of radiation-curable oligomers containing at least one terminal linear moiety can also improve the efficiency of the inner primary coating to transmit the stripping force applied during the ribbon stripping operation.

Examples of radiation-curable oligomers according to the present invention that provide enhanced strippability can be illustrated by the following formula:



wherein

- R<sup>4</sup> is a substantially linear long chain alkyl terminating in at least one hydroxyl group;
- each L represents, independently, a molecular bridging group, preferably derived from a diisocyanate precursor reactant;
- each x represents a resulting reacted linking group, such as, inter alia, a urethane, thio-urethane, or urea entity.

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Alternatively, ester linkages can also be utilized;  
R<sup>5</sup> is a linear or a branched or cyclic hydrocarbon or polyether moiety derived from a diol and having a molecular weight of from 150 to 10,000, preferably from 500 to 5,000, and most preferably from 1,000 to 2,000 Daltons;

R<sup>6</sup> is an end group carrying a radiation-curable functional group as defined herein, preferably an acrylate or methacrylate, and also having an hydroxyl linkage to the L entity.

R<sup>4</sup> preferably has at least about 80%, more preferably at least about 90%, of its carbon atoms in a straight chain; and,

n may represent a number from zero to 30.

Preferably, R<sup>4</sup> is an alkyl radical with of from about C<sub>9</sub> to about C<sub>20</sub>, since longer carbon chains may decrease the resistance against oil. Suitable examples of alkyls are lauryl, decyl, isodecyl, tridecyl, and stearyl. Most preferred is lauryl.

R<sup>5</sup> can contain a branched or cyclic aliphatic group having about 6 to about 15 carbon atoms. In particular R<sup>5</sup> can be the aliphatic component of a diisocyanate compound such as isophorone diisocyanate, DesW, TMDI, and HXDI. If R<sup>5</sup> is a branched component, preferably, the extent of branching units is at least about 10 mole %, and more preferably at least about 20 mole %, based on the total number of carbon atoms in R<sup>5</sup>.

The oligomers according to above formula can be made, for example, by reacting in a first reaction one mole of a diisocyanate compound (for forming R<sup>5</sup>) with (1) one mole of a long chain alkyl containing a hydroxy group (for forming R<sup>4</sup>) or (2) one mole of a compound containing a hydroxy functional group and a radiation-curable functional group (for forming R<sup>6</sup>). The urethane linking group “x” attached to “L” is formed by the reaction of the isocyanate group with a hydroxyl group. In a second reaction, the remaining isocyanate group is reacted with the other as yet unreacted hydroxyl group of the compound. Reactions of hydroxy functional compounds with isocyanate functional molecules are well known in the art, and can be catalyzed if needed, with known catalysts. Suitable examples of reactants containing a radiation-curable functional group and a hydroxy group are hydroxyethylacrylate or 2-hydroxypropylacrylate. Suitable examples of linear long chain alkyls include lauryl alcohol, decyl alcohol, isodecyl alcohol, tridecyl alcohol, and stearyl alcohol.

The resulting radiation-curable oligomer can be used in optical glass fiber coatings, in particular in inner primary coatings, as a monomer that enhances the strippability of the final coating, and that yields a coating composition which may have a high cure speed.

The radiation-curable oligomers according to this invention can accordingly be used in amounts suitable to provide the desired level of ribbon stripping performance. The desired amount can easily be determined by one skilled in the art by simple testing of different amounts of the selected linear oligomer(s) in an inner primary coating, and optionally in an outer primary coating as well, on optical glass fibers encased in a ribbon assembly. It has been found that the oligomers provided by this invention can generally be used in amounts of about 1 to about 90 wt. %, preferably about 5 to about 80 wt. %, and most preferably about 5 to about 60 based on the total weight of the inner primary or outer primary composition.

Example 8-1 and Comparative Examples H-1 Through H-3

The components shown in Table 13 were combined to inner primary coating compositions.



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75 micron thick films of the coating materials were prepared and suitably cured. The fiber pull-out friction test was conducted, as described herein, and the test results are shown in Table 13.

TABLE 13

Component (Amount is % by weight of total composition)	Comp. Example H-1	Comp. Example H-2	Comp. Example H-3	Example 8-1
Oligomer H- (I-PTHF2000) <sub>2</sub> -I-H	52.26	52.26	52.56	52.56
Ethoxylated Nonylphenol Acrylate	15.67	15.67	15.67	15.67
Lauryl Acrylate	3.39	10.79	10.79	7.15
n-Vinyl Formamide	23.6	0	0	0
Isobornyl Acrylate				
n-Vinyl Formamide	0	16.2	21.6	0
Ethylhexyl Acrylate				
n-Vinyl Formamide	0	0	0	19.84
Butyl Acrylate				
25:75 weight/weight of Bis(2,6-Dime- thoxybenzoyl) (2,4,4- Trimethylpentyl) Phosphine Oxide and 2-Hydroxy-2-Methyl-1- Phenyl Propanone gamma-Mercaptopropyl Trimethoxy Silane	3.7	3.7	3.7	3.7
Thioethylene Bis (3,5 di-tert- butyl-4-hydroxyl) Hydrocinnamate (antioxidant)	0.92	0.92	0.92	0.92
Test Results				
Fiber Pull-Out	0.46	0.46	0.46	0.46
Residue Test				
	1.5	0.75	1	0.65

The oligomer was formed by reacting the following components:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
PTHF2000 = 2000 molecular weight Polytetramethylene Ether Glycol (BASF)

The test results in Table 13 demonstrate that as the length of the linear moiety is increased, the fiber pull-out friction decreases.

Aromatic Groups

Ribbon strippability can also be enhanced by incorporating a high concentration of aromatic groups in the oligomers and monomers used to form the inner primary coating. It will be appreciated that coating compositions comprising about 0.1 or more moles of aromatic groups per 100 grams of total composition, calculated using the molecular weights of the compositional components, are regarded as having a high concentration of aromatic groups. It is believed that the planarity of the phenyl ring next to the surface of the optical glass fiber may allow for the good slidability of the inner primary coating off the optical glass fiber during ribbon stripping.

Example 9-1

The components shown in Table 14 were combined to form an inner primary coating composition.

A 75 micron thick film of the coating material was prepared and suitably cured. The crack propagation was then measured. A friction test was also conducted, as described herein. The results are shown in Table 14.

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TABLE 14

Component (Amount is % by weight of total composition)	Example 9-1
Oligomer H-I-(PTGL2000-I) <sub>2</sub> -H	51.54
Ethoxylated Nonylphenol Acrylate	20.86
Phenoxyethyl Acrylate	16.8
Lauryl Acrylate	7
25:75 weight/weight of Bis(2,6- Dimethoxybenzoyl) (2,4,4- Trimethylpentyl) Phosphine Oxide and 2- Hydroxy-2 -Methyl-1-Phenyl Propanone	2.5
Thiodiethylene Bis (3,5-di-tert-butyl- gamma-hydroxy) Hydrocinnamate	0.3
gamma-Mercaptopropyl Trimethoxy Silane	1
Test Results	
Crack Propagation (mm)	1.49
Fiber Pull-Out Friction (g/mm)	10
Predicted Strip Cleanliness	2

The oligomer was formed by reacting the following components:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
PTGL2000 = 2000 molecular weight polymethyltetrahydrofurfuryl/  
polytetrahydrofurfuryl copolymer diol (Mitsui, NY)

High Molecular Weight Polymeric Blocks and Reduced Concentration of Urethane

Radiation-curable, inner primary optical glass fiber coating compositions (hereinafter referred to as "inner primary compositions") are now well known in the art. Such inner primary compositions usually contain at least one radiation-curable oligomer, and optionally reactive diluents, photoinitiators, and additives, as described herein above.

It has now been found that by reformulating the radiation-curable oligomer used in the inner primary composition, an inner primary coating can be formed having a significantly increased crack propagation in combination with a significantly decreased fiber friction. Furthermore, it has been found that the crack propagation can be increased and the fiber friction decreased to levels which provide the inner primary coating with the ability to strip cleanly from the surface of an optical glass fiber during ribbon stripping, without the use of substantial amounts of slip agents in the inner primary coating. In some instances, the use of slip agents can be substantially avoided. The term slip agents includes components which are separate and distinct from the radiation-curable oligomer as well as slip agent moieties that can be bound to the radiation-curable oligomer. The use of slip agents may cause undesirable delamination of the inner primary coating during use of the ribbon assembly in hot and wet environments, such as tropical environments, which can lead to microbending and attenuation of the signal transmission. Thus, by substantially avoiding the use of slip agents to provide a ribbon-strippable inner primary coating, the present invention can provide a ribbon-strippable inner primary coating which exhibits enhanced resistance to such undesirable delamination.

Radiation-curable, oligomers comprising a carbon containing backbone to which at least one radiation-curable functional group is bound are well known in the art. Usually, the carbon containing backbone of the radiation-curable oligomer contains one or more polymeric blocks each having a molecular weight up to about 2000 and being connected together via coupling groups. Thus, an oligomer having a molecular weight of about 6000, will usually contain three polymeric blocks each having a molecular weight of about 2000 which are connected via coupling groups. The radiation-curable functional groups are also usually connected to the carbon-containing backbone via coupling groups.

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By extensive experimentation, it has now been found that as the molecular weight of the polymeric blocks is increased, the crack propagation of the inner primary coating increases and the fiber friction of the inner primary coating decreases. The molecular weight of the polymeric blocks should be adjusted up to level which provides an inner primary coating having a fiber friction and crack propagation that are suitable for ribbon stripping. For example, the molecular weight of the polymeric block can be adjusted upward to level which provides an inner primary coating having a combination of fiber friction and crack propagation that provides a predicted strip cleanliness of about 3 or less, and preferably about 2 or less. Alternatively, the molecular weight of the polymeric block can be adjusted upward to level which provides an inner primary coating having a fiber friction of about 30 g/mm or less at a rate of 0.1 mm/sec in combination with a crack propagation of at least about 1.3 mm at a rate of 0.1 mm/sec, at a ribbon stripping temperature. Preferably, the fiber friction is about 25 g/mm or less and more preferably about 20 g/mm or less. Preferably, the crack propagation is at least about 1.5 mm and more preferably at least about 2 mm. The crack propagation is usually below about 4, but can be higher.

It has been found that by using polymeric blocks having a molecular weight greater than 2000, preferably at least about 2500, and most preferably at least about 3000, inner primary coatings having a fiber friction and a crack propagation as described above can be provided. The molecular weight of said polymeric block is usually less than about 10,000, preferably less than about 8,000.

The coupling groups can be any group capable of providing a link between polymer blocks and/or between radiation-curable functional groups and polymer blocks. Examples of suitable coupling groups are urethane, urea and thiourethane. For purposes of practicing the present invention, which relates to adjusting the crack propagation and fiber friction using the molecular weight of the polymeric blocks and/or urethane concentration, the following groups are not considered coupling groups when determining the molecular weight of the polymeric blocks: carbonate, ether, and ester groups. Thus, when determining the molecular weight of the polymeric block, ether groups, carbonate groups, and ester groups are considered part of the polymeric block. Polymeric compounds separated by urethane, thiourethane and urea groups are considered separate polymeric blocks. Urethane is the preferred coupling group.

Usually, urethane groups are used as the coupling groups in the radiation-curable oligomer. For example, if an oligomer having a number average molecular weight about 6000 comprising 3 polymer blocks, each having a number average molecular weight of about 2000, and containing 2 radiation-curable functional groups, will have four urethane linkages. Two of the urethane linkages connect the radiation-curable groups to the polymeric blocks and two of the urethane linkages connect the three polymeric blocks together.

It has now been found that as the concentration of urethane linkages present in the inner primary composition is decreased, the crack propagation of the inner primary coating increases and the fiber friction of the inner primary coating decreases. Thus, the term urethane concentration represents the weight percentage of all urethane linkages present in the inner primary coating composition, based on the total weight of the inner primary coating composition.

Based on this discovery, the urethane concentration should be adjusted downward to a level which provides an inner primary coating having a fiber friction and crack propagation that are suitable for ribbon stripping the desired

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ribbon assembly. For example, the urethane concentration can be adjusted downward to a level which provides an inner primary coating having a combination of fiber friction and crack propagation that provides a predicted strip cleanliness of about 3 or less, and preferably about 2 or less. It has been found that if the concentration of urethane linkages is about 4% by weight or less, inner primary coatings having a fiber friction and a crack propagation that exhibit a predicted strip cleanliness of about 3 or less can be provided. Preferably, the urethane concentration is about 3.5% by weight or less, more preferably about 2.5% or less by weight, and most preferably about 2% or less by weight. The urethane concentration effect on fiber friction and crack propagation is more pronounced for higher molecular weight oligomers, such as about 3,000 to about 10,000, more preferably about 3,500 to about 8,000. Thus, preferably the urethane oligomer has a molecular weight of about 3,000 to about 10,000 in combination with a urethane concentration of about 4% by weight or less, more preferably, a molecular of about 3,500 to about 8,000 in combination with a urethane concentration of about 3.5% or less, and most preferably, a molecular weight of about 3,500 to about 8,000 in combination with a urethane concentration of about 3% or less.

The polymeric blocks can comprise for example polyethers, polyolefins, polycarbonates, polyesters, polyamides or copolymers thereof. Preferably, the polymeric blocks comprise polyethers.

The radiation-curable functional groups used can be any functional group capable of polymerization when exposed to actinic radiation. Suitable radiation-curable functional groups are now well known and within the skill of the art.

Commonly, the radiation-curable functionality used is ethylenic unsaturation, which can be polymerized through radical polymerization or cationic polymerization. Specific examples of suitable ethylenic unsaturation are groups containing acrylate, methacrylate, styrene, vinyl ether, vinyl ester, N-substituted acrylamide, N-vinyl amide, maleate esters, and fumarate esters. Preferably, the ethylenic unsaturation is provided by a group containing acrylate, methacrylate, or styrene functionality, and most preferably acrylate or methacrylate.

Another type of radiation-curable functionality generally used is provided by, for example, epoxy groups, or thiol-ene or amine-ene systems. Epoxy groups can be polymerized through cationic polymerization, whereas the thiol-ene and amine-ene systems are usually polymerized through radical polymerization. The epoxy groups can be, for example, homopolymerized. In the thiol-ene and amine-ene systems, for example, polymerization can occur between a group containing allylic unsaturation and a group containing a tertiary amine or thiol.

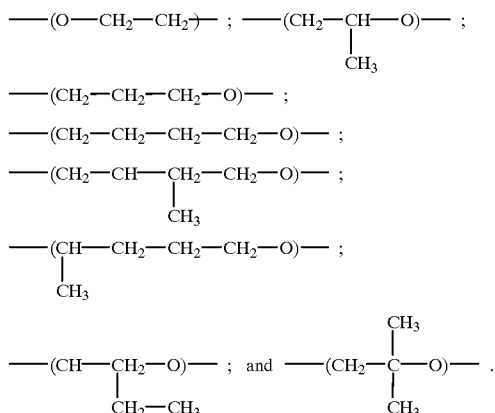
The radiation-curable oligomer can be easily formed by reacting a polymeric polyol, a compound containing a radiation-curable functional group and a hydroxyl group, and a polyisocyanate. The general reaction of isocyanate functional groups with hydroxyl groups to form urethane linkages is well known in the art. Thus, one skilled in the art will be able to make the improved oligomer according to the present invention based on the disclosure provided herein.

Examples of suitable polymeric polyols that can be used to form the radiation-curable oligomer include polyether diols, polyolefin diols, polyester diols, polycarbonate diols, and mixtures thereof. Polyether and polycarbonate diols, or combinations thereof, are preferred. The polymeric block is the residue of the polymeric polyol after reaction to form the radiation-curable oligomer.

If a-polyether diol is used, preferably the polyether is a substantially non-crystalline polyether. Preferably, the poly-

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ether comprises repeating units of one or more of the following monomer groups:



Thus, suitable polyethers can be made from epoxy-ethane, epoxy-propane, tetrahydrofuran, methyl-substituted tetrahydrofuran, isobutylene, and the like. Commercial examples of a suitable polyether polyols that can be used are PTGL2500, PTGL3000, PTGL3500, and PTGL4000 (Hodogaya Chemical Company).

If a polyolefin diol is used, the polyolefin is preferably a linear or branched hydrocarbon containing a plurality of hydroxyl end groups. The hydrocarbon provides a hydrocarbon backbone for the oligomer. Preferably, the hydrocarbon is a non-aromatic compound containing a majority of methylene groups ( $\text{—CH}_2\text{—}$ ) and which can contain internal unsaturation and/or pendant unsaturation. Examples of suitable hydrocarbon diols include, for example: hydroxyl-terminated; fully or partially hydrogenated 1,2-polybutadiene; copolymers of 1,4-polybutadiene; copolymers of 1,2-polybutadiene; polyisobutylene polyol; mixtures thereof, and the like. Preferably, the hydrocarbon diol is a substantially, fully hydrogenated 1,2-polybutadiene-ethene copolymer or 1,2-polybutadiene-ethene copolymer.

Examples of polycarbonate diols are those conventionally produced by the alcoholysis of diethylene carbonate with a diol.

Examples of polyester diols include the reaction products of saturated polycarboxylic acids, or their anhydrides, and diols. Commercial examples are the polycaprolactones, commercially available from Union Carbide under the trade designation Tone Polyol series of products, for example, Tone 0200, 0221, 0301, 0310, 2201, and 2221. Tone Polyol 0301 and 0310 are trifunctional.

Any organic polyisocyanate, alone or in admixture, can be used as the polyisocyanate. Examples of suitable diisocyanates include: isophorone diisocyanate (IPDI); toluene diisocyanate (TDI); diphenylmethylene diisocyanate; hexamethylene diisocyanate; cyclohexylene diisocyanate; methylene dicyclohexane diisocyanate; 2,2,4-trimethyl hexamethylene diisocyanate; m-phenylene diisocyanate; 4-chloro-1,3-phenylene diisocyanate; 4,4'-biphenylene diisocyanate; 1,5-naphthylene diisocyanate; 1,4-tetramethylene diisocyanate; 1,6-hexamethylene diisocyanate; 1,10-decamethylene diisocyanate; 1,4-cyclohexylene diisocyanate; and polyalkyloxide and polyester glycol diisocyanates such as polytetramethylene ether glycol terminated with TDI and

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polyethylene adipate terminated with TDI, respectively. Preferably, the isocyanates are TDI or IPDI.

If other oligomers, monomers, and/or additives containing urethane linkages are used in admixture with the above described radiation-curable oligomer to form an inner primary composition, the concentration of urethane linkages present in each other oligomer, monomer or additive should be included in the urethane concentration calculation. Examples of common monomers containing urethane linkages include:

trimethyl-olpropane triacrylate,  
the triacrylate or methacrylate from hexane-2,4,6 triol, or  
from glycerol, ethoxylated glycerol, or propoxylated  
glycerol,

hexanediol diacrylate,  
1,3-butylene glycol diacrylate,  
neopentyl glycol diacrylate,  
1,6-hexanediol diacrylate,  
20 neopentyl glycol diacrylate,  
polyethylene glycol-200 diacrylate,  
tetraethylene glycol diacrylate,  
triethylene glycol diacrylate,  
25 pentaerythritol tetraacrylate,  
tripropylene glycol diacrylate,  
ethoxylated bisphenol-A diacrylate,  
trimethylolpropane diacrylate,  
di-trimethylolpropane tetraacrylate,  
30 triacrylate of tris(hydroxyethyl) isocyanurate,  
dipentaerythritol hydroxypentaacrylate,  
pentaerythritol triacrylate,  
ethoxylated trimethylolpropane triacrylate,  
35 triethylene glycol dimethacrylate,  
ethylene glycol dimethacrylate,  
tetraethylene glycol dimethacrylate,  
polyethylene glycol-2000 dimethacrylate,  
1,6-hexanediol dimethacrylate,  
40 neopentyl glycol dimethacrylate,  
polyethylene glycol-600 dimethacrylate,  
1,3-butylene glycol dimethacrylate,  
ethoxylated bisphenol-A dimethacrylate, trimethylolpro-  
pane trimethacrylate,  
45 diethylene glycol dimethacrylate,  
1,4-butanediol diacrylate,  
diethylene glycol dimethacrylate,  
pentaerythritol tetramethacrylate,  
50 glycerin dimethacrylate,  
trimethylolpropane dimethacrylate,  
pentaerythritol trimethacrylate,  
pentaerythritol dimethacrylate,  
pentaerythritol diacrylate, and  
55 the like and mixtures thereof.

60 Mono(meth)acrylates such as cyclohexyl(meth)acrylate,  
isobornyl(meth)acrylate,  
lauryl(meth)acrylate,  
alkoxylated phenolacrylate,  
isooctyl-acrylate,  
2-ethylhexyl-acrylate,  
hydroxyethyl acrylate, and  
tetrahydrofurfuryl(meth)-acrylate.

65 The Invention will be further explained by the following non-limiting examples illustrating the use of block polymeric formulations.

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Examples 10-1 Through 10-14 and Comparative Examples J-1 Through J-11

Inner primary compositions were made by combining the components shown in Tables 15 and 16, in the same manner as described herein above. The viscosity of the compositions

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was measured as described above, and the results are shown in Tables 15 and 16. The inner primary compositions were cured by exposure to UV radiation and the fiber friction and crack propagation properties were measured, in the same manner as described herein above. The test results are shown in Tables 15 and 16.

TABLE 15

Component (% by weight of total composition)	Ex. 10-1	Ex. 10-2	Ex. 10-3	Ex. 10-4	Ex. 10-5	Ex. 10-6	Ex. 10-7
Oligomer H-I-PPG3025-I-H	49.38	0	0	0	0	0	0
Oligomer H-I-PPG4025-I-H	0	49.38	0	0	0	0	0
Oligomer H-I-PTGL3000-I-H	0	0	64.38	0	0	49.38	0
Oligomer H-I-PTGL3500-I-H	0	0	0	64.38	0	0	49.38
Oligomer H-I-PTGL4000-I-H	0	0	0	0	64.38	0	0
Oligomer H-(I-PTGL2000) <sub>2</sub> -I-H	0	0	0	0	0	0	0
Phenoxy Ethyl Acrylate	0	0	0	0	0	0	0
Ethoxylated Nonylphenol Acrylate Ester	40.32	40.32	25.32	25.32	25.32	40.32	40.32
Isobornyl Acrylate	0	0	0	0	0	0	0
Lauryl Acrylate	6	6	6	6	6	6	6
Thiodiethylene bis(3,5-di-tert-butyl-4-Hydroxy)Hydrocinnamate	.3	.3	.3	.3	.3	.3	.3
2,4,6-Trimethylbenzoyl Diphenyl Phosphine Oxide and 2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone	3	3	3	3	3	3	3
2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone Bis(2,6-Dimethoxybenzyl)(2,4,4-Trimethylphenyl)-Phosphine Oxide	0	0	0	0	0	0	0
gamma-Mercaptopropyl Trimethoxy Silane	1	1	1	1	1	1	1
Test Results							
Viscosity mPa · s (25 C.)	1200	1190	25350	321350	16840	9380	17680
Urethane Concentration (%) <sup>1</sup>	2.01	1.76	2.99	2.65	2.24	2.29	2.03
Fiber Pull-Out Friction (g/mm)	19.3	10.8	23.23	21.27	27.63	21.84	14.04
Crack Propagation (mm)	1.5	2.18	1.66	1.63	1.82	1.76	2.32

Component (% by weight of total composition)	Ex. 10-8	Ex. 10-9	Ex. 10-10	Ex. 10-11	Ex. 10-12	Ex. 10-13	Ex. 10-14
Oligomer H-I-PPG3025-I-H	0	0	0	0	0	0	0
Oligomer H-I-PPG4025-I-H	0	0	0	0	0	0	0
Oligomer H-I-PTGL3000-I-H	0	0	0	0	0	0	0
Oligomer H-I-PTGL3500-I-H	0	0	39.38	45.23	45.23	65.33	64.68
Oligomer H-I-PTGL4000-I-H	49.38	0	0	0	0	0	0
Oligomer H-(I-PTGL2000) <sub>2</sub> -I-H	0	50.54	0	0	0	0	0
Phenoxy Ethyl Acrylate	0	14	0	25.13	20.1	20.1	17.91
Ethoxylated Nonylphenol Acrylate Ester	40.32	15.16	50.21	25.13	20.1	0	0
Isobornyl Acrylate	0	10	0	0	10.5	10.5	6.97
Lauryl Acrylate	6	6	5.99	0	0	0	5.97
Thiodiethylene bis(3,5-di-tert-butyl-4-Hydroxy)Hydrocinnamate	.3	.3	.3	.3	.3	.3	.3
2,4,6-Trimethylbenzoyl Diphenyl Phosphine Oxide and 2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone	3	0	0	0	0	0	0
2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone Bis(2,6-Dimethoxybenzyl)(2,4,4-Trimethylphenyl)-Phosphine Oxide	0	3	3	3	3	3	3
gamma-Mercaptopropyl Trimethoxy Silane	1	1	1	1	1	1	
Test Results							
Viscosity mPa · s (25 C.)	7050	5570	4530	7220	5540	10670	9440
Urethane Concentration (%) <sup>1</sup>	1.72	2.67	1.62	1.86	1.86	2.69	2.66
Fiber Pull-Out Friction (g/mm)	16.15	25.3	13.4	15.8	15.2	25.6	21.6
Crack Propagation (mm)	2.31	2.04	2.6	2	2.9	1.6	1.8

<sup>1</sup>The urethane concentration can be calculated from (1) the amount of NCO present in the urethane linkages of the oligomer, (2) the molecular weight of the oligomer, which can be measured by conventional methods, and (3) the amount of oligomer in the composition. The polyol molecular weights in Table I are estimated rather than measured molecular weights.

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TABLE 16

Component (% by weight based on total weight of composition)	Comp. Ex. J-1	Comp Ex. J-2	Comp Ex. J-3	Comp Ex. J-4	Comp Ex. J-5	Comp Ex. J-6	Comp Ex. J-7	Comp Ex. J-8	Comp Ex. J-9	Comp. Ex. J-10	Comp. Ex. J-11
Oligomer H-I-PTGL2000-I-H	0	83.7	68.7	79.7	79.7	74.7	69.7	67.7	79.7	49.38	64.38
Oligomer H-(I-PERM1000) <sub>1,4</sub> -(I-PPG1025) <sub>1,06</sub> -I-H	56	0	0	0	0	0	0	0	0	0	0
isodecyl acrylate	14										
HI	0	0	0	0	0	5	10	15	0	0	0
Phenoxy Ethyl Acrylate		0	0	5	0	10	10	7	0	0	0
Ethoxylated Nonylphenol Acrylate Ester	25.5	6	21	0	0	0	0	0	0	40.32	25.32
Isobornyl Acrylate	0	0	0	5	10	0	0	0	10	0	0
Lauryl Acrylate	0	6	6	6	6	6	6	6	6	6	6
Thiodiethylene bis(3,5-di-tert-butyl-4-Hydroxy)Hydrocinnamate	.5	.3	.3	.3	.3	.3	.3	.3	.3	.3	.3
2,4,6-Trimethylbenzoyl Diphenyl Phosphine Oxide and 2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone	0	3	3	3	3	3	3	3	0	3	3
2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone Bis(2,6-Dimethoxybenzyl)	3	0	0	3	3	3	3	3	3	0	0
(2,4,4-Trimethylpentyl)Phosphine Oxide											
gamma-Mercaptopropyl Trimethoxy Silane	1	1	1	1	1	1	1	1	1	1	1
Test Results											
Viscosity mPa · s (25 C.)	6000									5050	10310
Urethane Concentration (%)	4.9	5.38	4.42	5.12	5.12	5.31	5.49	5.87	5.12	3.53	4.30
Fiber Pull-Out Friction (g/mm)	44	41.97	34.84	41.8	40.2	44.6	40.4	39.9	44.9	34.84	41.17
Crack Propagation (mm)	1	1.32	1.45	1.21	1.25	1.05	1.2	1.17	1.2	1.45	1.32

The oligomers in Tables 15 and 16 were formulated from the following components:  
H = Hydroxyethylacrylate  
I = Isophoronediiisocyanate  
HI = Hexane diisocyanate  
PTGL2000 = polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl copolymer diol having the molecular weight of 2000 (Mitsui, NY)  
PPG1025 = polypropyleneoxide diol having an average molecular weight of 1025, (BASF)  
Perm 1000 = Permanol KM10-1733 polycarbonate/polyether copolymer diol having an average molecular weight of 1000

As can be seen from Examples 10-6 through 10-8, and Comparative Example J-10, as the concentration of urethane decreases, the crack propagation increases and the fiber friction decreases. Furthermore, as the molecular weight of the polymeric blocks increases, the crack propagation increases and the fiber friction decreases.

Similarly, as can be seen from Examples 10-3 through 10-5, and Comparative Example J-11, as the concentration of urethane decreases, the crack propagation increases and the fiber friction decreases. Furthermore, as the molecular weight of the polymeric blocks increases, the crack propagation increases and the fiber friction decreases. Examples 10-3 through 10-5 used significantly more of the radiation-curable oligomer than Examples 10-6 through 10-8, and the same trend in fiber friction and crack propagation was clearly demonstrated. Based on this experimental evidence, the trend in fiber friction and crack propagation is dependent mainly upon the oligomer. Furthermore, these Examples used a polyether polymeric block.

Examples 10-1 and 10-2 demonstrate that when a polypropylene oxide polymeric block is used as a polyether polymeric block, the crack propagation and fiber friction are still dependent upon the molecular weight of the polymeric block and/or the concentration of urethane if used. In particular, as the concentration of urethane decreases, the crack propagation increases and the fiber friction decreases. Furthermore, as the molecular weight of the polymeric blocks increases, the crack propagation increases and the fiber friction decreases.

Examples 10-4, 10-7 and 10-10 through 10-14 used different amounts of the same radiation-curable oligomer.

The experimental results demonstrate that the trend in crack propagation and fiber friction is based on the molecular weight of the polymeric block and/or the concentration of urethane.

The test results in Table 15 demonstrate that the above described trends regarding crack propagation and fiber friction based on molecular weight and/or urethane concentration are independent of the type of oligomers and is generally consistent among the different types of oligomers.

FIG. 7 illustrates a graph which includes the data shown in Tables 15 and 16, above. As can be seen from FIG. 7, the urethane concentration in the inner primary composition directly affects the fiber pull-out friction. As the urethane concentration is decreased, the fiber pull-out friction is decreased.

FIG. 8 illustrates a graph of the fiber friction versus urethane concentration for Examples 10-10 through 10-14. FIG. 8 clearly demonstrates the direct correlation between fiber pull-out friction and urethane concentration in the inner primary composition. In particular, as the urethane concentration decreases the fiber pull-out friction decreases.

Examples 10-15 Through 10-22

Inner primary compositions were made by combining the components shown in Table 17, in the same manner as described herein above. The viscosity of the compositions was measured as described above, and the results are shown in Table 17. The inner primary compositions were cured by exposure to UV radiation and the fiber friction and crack propagation properties were measured, in the same manner as described herein above. The test results are shown in Table 17.

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TABLE 17

Component (% by weight based on total weight of composition)	Exmp. 10-15	Exmp. 1-16	Exmp. 10-17	Exmp. 10-18	Exmp. 10-19	Exmp. 10-20	Exmp. 10-21	Exmp. 10-33
Oligomer H-(I-PTGL2000) <sub>2</sub> -I-H	23.71	28.45	33.19	37.93	42.68	47.42	52.16	56.9
Phenoxy Ethyl Acrylate	64.68	58.52	52.35	46.19	40.03	33.86	27.7	21.53
Ethoxylated Nonylphenol Acrylate Ester	7.11	8.53	9.95	11.38	12.87	14.22	15.64	17.06
Thiodiethylene bis(3,5-di-tert-butyl-4-Hydroxy)Hydrocinnamate	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone Bis(2,6-Dimethoxybenzyl)(2,4,4-Trimethylpentyl)Phosphine Oxide	3	3	3	3	3	3	3	3
gamma-Mercaptopropyl Trimethoxy Silane	1	1	1	1	1	1	1	1
Test Results								
Viscosity mPa · s (25 C.)	430	730	1200	2000	3270	5070	8500	14,500
Urethane Concentration (%)	1.25	1.5	1.75	2	2.25	2.5	2.75	3
Fiber Pull-Out Friction (g/mm)	20.5	22.7	23.9	28.7	26.4	35.2	31.8	40.1
Crack Propagation (mm)	2.31	2.32	1.96	1.89	1.8	1.77	1.75	1.51

The oligomers in Table 17 were formulated from the following components:  
H = Hydroxyethylacrylate  
I = Isophoronediiisocyanate  
PTGL 2000 = polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl copolymer diol having an average molecular weight of 2000, (Mitsui, NY)

FIG. 9 illustrates a graph of the experimental results of Examples 10-15 through 10-22. As can be seen from FIG. 9, as the urethane concentration decreases the fiber pull-out friction decreases.

Examples 10-22A Through 10-24

Inner primary compositions were made by combining the components shown in Table 18, in the same manner as described herein above. The viscosity of the compositions was measured as described above, and the results are shown in Table 18. The inner primary compositions were cured by exposure to UV radiation and the fiber friction and crack propagation properties were measured, in the same manner as described herein above. The test results are shown in Table 18.

Table 18

Component (% by weight based on total weight of composition)	Example 10-22A	Example 10-23	Ex-ample 10-24
Oligomer H-I-PTGL4200-I-H	0	49.38	45.01
Oligomer H-(I-PTGL2000) <sub>2</sub> -I-H	49.38	0	0
Monomer H-HI	0	0	8.85
Ethoxylated Nonylphenol Acrylate Ester	40.32	40.32	36.75
Thiodiethylene bis(3,5-di-tert-butyl-4-Hydroxy) Hydrocinnamate	0.3	0.3	0.27
Lauryl Acrylate	6	6	5.47
2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone Bis (2,6-Dimethoxybenzyl) (2,4,4-Trimethylpentyl) Phosphine Oxide	3	3	2.73
gamma-Mercaptopropyl Trimethoxy Silane	1	1	.91
Test Results			
Viscosity mpa.s (25° C.)	9260	7050	5100
Urethane Concentration (%)	2.61	1.72	2.61
Fiber Pull-Out Friction (g/mm)	20.85	16.85	17.4
Crack Propagation (mm)	1.62	2.06	1.74

The oligomers and monomers in Table 18 were formulated from the following components:  
H = Hydroxyethylacrylate  
I = Isophoronediiisocyanate  
HI = Hexane isocyanate

Table 18-continued

Component (% by weight based on total weight of composition)	Example 10-22A	Example 10-23	Ex-ample 10-24
PTGL 4200 = polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl copolymer diol having an average molecular weight of 4200, (Mitsui, NY) PTGL 2000 = polymethyltetrahydrofurfuryl/polytetrahydrofurfuryl copolymer diol having an average molecular weight of 2000, (Mitsui, NY)			

By comparing Example 10-22A with 10-24, it becomes clear that by using a higher molecular weight polymeric block, 4200 g/mole in Example 10-24 compared to 2000 g/mole in Example 10-22A, the fiber friction can be significantly decreased and the crack propagation can be increased. The urethane concentration was the same for both Examples 10-22A and 10-24. The oligomer used contained a polyether backbone.

Examples 10-25 Through 10-28

Inner primary compositions were made by combining the components shown in Table 19, in the same manner as described herein above. The viscosity of the compositions was measured as described above, and the results are shown in Table 19. The inner primary compositions were cured by exposure to UV radiation and the fiber friction and crack propagation properties were measured, in the same manner as described herein above. The test results are shown in Table 19.

TABLE 19

Component (% by weight based on total weight of composition)	Ex. 10-25	Ex. 10-26	Ex. 10-27	Ex. 10-28
Oligomer H-I-(NissoPB2000) <sub>2</sub> -I-H	50	45	40	35
Ethoxylated Nonylphenol Acrylate Ester	29.5	34.5	39.5	44.5
Isobornyl Acrylate	10	10	10	10
Thiodiethylene bis(3,5-di-tert-butyl-4-Hydroxy) Hydrocinnamate	0.5	0.5	0.5	0.5
Lauryl Acrylate	6	6	6	6
2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone Bis (2,6-Dimethoxybenzyl) (2,4,4-Trimethylpentyl) Phosphine Oxide	3	3	3	3

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TABLE 19-continued

Component (% by weight based on total weight of composition)	Ex. 10-25	Ex. 10-26	Ex. 10-27	Ex. 10-28
gamma-Mercaptopropyl Trimethoxy Silane	1	1	1	1
Test Results				
Viscosity mPa.s (25° C.)				
Urethane Concentration (%)	2.64	2.37	2.11	1.84
Fiber Pull-Out Friction (g/mm)	13.88	13.49	9.24	6.94
Crack Propagation (mm)	1.79	1.52	*	*

The oligomers and monomers in Table 19 were formulated from the following components:  
H = Hydroxyethylacrylate  
I = Isophoronediiisocyanate  
NissoPB 2000 = Polybutadiene copolymer diol having an average molecular weight of 2000, (Nippon Soda)  
\*The crack propagation could not be measured for these two coatings.

FIG. 10 illustrates a graph of the experimental results of Examples 10-25 through 10-28. As can be seen from FIG. 10, as the urethane concentration decreases the fiber pull-out friction decreases. The oligomer used contained a polyolefin backbone.

Examples 10-29 Through 10-32

Inner primary compositions were made by combining the components shown in Table 20 in the same manner as described herein above. The viscosity of the compositions was measured as described above, and the results are shown in Table 20. The inner primary compositions were cured by exposure to UV radiation and the fiber friction and crack propagation properties were measured, in the same manner as described herein above. The test results are shown in Table 20.

TABLE 20

Component (% by weight based on total weight of composition)	Ex. 10-29	Ex. 10-30	Ex. 10-31	Ex. 10-32
Oligomer H-I-PTGL2000-I-H	40	40	40	40
H-BI	0	4.24	9.18	14.12
Ethoxylated Nonylphenol Acrylate Ester	10	10	10	10
Phenoxethyl Acrylate	45.5	41.26	36.32	31.38
Thiodiethylene bis(3,5-di-tert-butyl-4-Hydroxy) Hydrocinnamate	0.5	0.5	0.5	0.5
2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone Bis(2,6-Dimethoxybenzyl)(2,4,4-Trimethylpentyl) Phosphine Oxide	3	3	3	3
gamma-Mercaptopropyl Trimethoxy Silane	1	1	1	1
Test Results				
Viscosity mpa.s (25° 0.C)	770	810	870	950
Urethane Concentration (%)	2.5	3	3.5	4
Fiber Pull-Out Friction (g/mm)	39.1	40.4	42.7	47.9
Crack Propagation (mm)	1.27	1.24	1.28	1.13

The oligomers and monomers in Table 20 were formulated from the following components:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
BI = Butyl Isocyanate

FIG. 11 illustrates a graph of the experimental results of Examples 10-29 through 10-32. As can be seen from FIG. 11, as the urethane concentration decreases the fiber pull-out friction decreases.

Examples 10-33 Through 10-36

Inner primary compositions were made by combining the components shown in Table 21, in the same manner as described herein above. The viscosity of the compositions was measured as described above, and the results are shown in Table 21. The inner primary compositions were cured by exposure to UV radiation and the fiber friction and crack propagation properties were measured, in the same manner as described herein above. The test results are shown in Table 21.

TABLE 21

Component (% by weight based on total weight of composition)	Ex. 10-33	Ex. 10-34	Ex. 10-35	Ex. 10-36	Comp. Ex. J-1
Oligomer H-I-PPG2025-I-Perm2000-I-H	50	45	40	35	0
Oligomer H-I-(PPG2025) <sub>1.4</sub> -I-(Perm1000) <sub>1.06</sub> -I-H	0	0	0	0	70
Ethoxylated Nonylphenol Acrylate Ester	10	10	10	10	25.5
Lauryl Acrylate	6	6	6	6	0
Thiodiethylene bis(3,5-di-tert-butyl-4-Hydroxy)Hydrocinnamate	0.5	0.5	0.5	0.5	0.5
2-Hydroxy-2-Methyl-1-Phenyl-1-Propanone Bix(2,6-Dimethoxybenzyl)(2,4,4-Trimethylphenyl)Phosphine Oxide	1.5	1.5	1.5	1.5	3
gamma-Mercaptopropyl Trimethoxy Silane	1	1	1	1	1
Test Results					
Viscosity mPa · s (25 C.)					6000
Urethane Concentration (%)	2.03	1.83	1.62	1.42	4.9
Fiber Pull-Out Friction (g/mm)	11.87	11.5	9.9	9.8	44
Crack Propagation (mm)	0	3.3	3.2	3.1	1

The oligomers and monomers in Table 21 were formulated from the following components:  
H = Hydroxyethyl Acrylate  
I = Isophorone Diisocyanate  
Perm 1000 = Permalon KM10-1733 polycarbonate/polyether copolymer diol having an average molecular weight of 1000  
PPG2025 = PC1122 a polycarbonate/polyether copolymer diol having an average molecular weight of 2000

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The test results in Table 21 illustrate that as the urethane concentration decreases the fiber pull-out friction decreases and the crack propagation increases. The test results in Table 21 also illustrate that as the molecular weight of the polymeric block increases the fiber pull-out friction decreases and the crack propagation increases. The polymeric block used was a polycarbonate.

#### Ribbon Assemblies

Ribbon assemblies are now well known in the art and one skilled in the art will readily be able to use the disclosure provided herein to prepare the novel ribbon assemblies having enhanced ribbon strippability for the desired applications. The novel ribbon assembly made according to this invention can be advantageously used in various telecommunication systems. Such telecommunication systems typically include ribbon assemblies containing optical glass fibers, in combination with transmitters, receivers, and switches. The ribbon assemblies containing the coated optical glass fibers are the fundamental connecting units of telecommunication systems. The ribbon assembly can be buried under ground or water for long distance connections, such as between cities. The ribbon assembly can also be used to connect directly to residential homes.

The novel ribbon assembly made according to this invention can also be used in cable television systems. Such cable television systems typically include ribbon assemblies containing optical glass fibers, transmitters, receivers, and switches. The ribbon assemblies containing the coated optical glass fibers are the fundamental connecting units of such cable television systems. The ribbon assembly can be buried under ground or water for long distance connections, such as between cities. The ribbon assembly can also be used to connect directly to residential homes.

The novel ribbon assemblies can also be used in a wide variety of technologies, including but not limited to, various security systems, data transmission lines, high density television, and computer appliance systems. It will be appreciated that as a result of the fundamental discoveries described herein including the relationship between the fiber friction forces and the cohesive strength of the coatings themselves, and the means to control and establish such features and functions, the optical fiber art is now able to realize significant advantages. These are primarily exhibited, as explained above, in the stripping and cable splicing function, but those operations are nonetheless critical in the establishment of a ribbon/cable network of communication.

While the invention has been described in detail and with reference to specific embodiments thereof, it will be apparent to those of ordinary skill in the art that various changes and modifications can be made to the claimed invention without departing from the spirit and scope thereof. For instance, while this invention has principally been described with reference to ribbon constructions and assemblies of optical fibers, it is equally adaptable to other geometric and structural arrays of multiple fiber conduits and cables.

Accordingly, applicants believe that the scope of this invention is defined solely by the terminology set forth in the following claims and is not otherwise limited.

What is claimed is:

1. A composition for coating an optical fiber, said composition comprising propoxylated nonyl phenol acrylate and an oligomer having at least one functional group capable of polymerizing under the influence of radiation, said composition after radiation cure having the combination of properties of:

- (a) a fiber pull-out friction of less than 40 g/mm at 90° C.;
- (b) a crack propagation of greater than 1.0 mm at 90° C.;

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- (c) a glass transition temperature of below 10° C.; and
- (d) sufficient adhesion to said glass fiber to prevent delamination in the presence of moisture and during handling.

2. A coated optical fiber comprising at least an inner primary coating and an outer primary coating, wherein said inner primary coating is obtained by curing the composition of claim 1.

3. A ribbon assembly comprising:

- (i) a plurality of coated optical fibers, at least one of said coated optical fibers being the coated optical fiber of claim 2; and

- (ii) a matrix material bonding said plurality of coated optical fibers together.

4. The composition of claim 1, wherein said oligomer comprises a polyether polyol residue, an isophorone diisocyanate residue, and a hydroxyethylacrylate residue.

5. The coated optical fiber of claim 2, wherein said oligomer comprises a polyether polyol residue, an isophorone diisocyanate residue, and a hydroxyethylacrylate residue.

6. The ribbon assembly of claim 3, wherein said oligomer comprises a polyether polyol residue, an isophorone diisocyanate residue, and a hydroxyethylacrylate residue.

7. The composition of claim 4, wherein said oligomer further comprises a bis 4,4'-(isocyanatocyclohexyl)methane residue.

8. The composition of claim 1, further comprising gamma-mercaptopropyl trimethoxysilane.

9. The composition of claim 7, further comprising gamma-mercaptopropyl trimethoxysilane.

10. A composition for coating an optical fiber, said composition comprising an oligomer having an aliphatic diisocyanate residue and at least one functional group capable of polymerizing under the influence of radiation, said composition after radiation cure having the combination of properties of:

- (a) a fiber pull-out friction of less than 40 g/mm at 90° C.;
- (b) a crack propagation of greater than 1.0 mm at 90° C.;
- (c) a glass transition temperature of -20° C. or less; and
- (d) sufficient adhesion to said glass fiber to prevent delamination in the presence of moisture and during handling.

11. A coated optical fiber comprising at least an inner primary coating and an outer primary coating, wherein said inner primary coating is obtained by curing the composition of claim 10.

12. A ribbon assembly comprising:

- (i) a plurality of coated optical fibers, at least one of said coated optical fibers being the coated optical fiber of claim 11; and

- (ii) a matrix material bonding said plurality of coated optical fibers together.

13. The composition of claim 10, wherein said oligomer further comprises a polyether polyol residue.

14. The coated optical fiber of claim 11, wherein said oligomer further comprises a polyether polyol residue.

15. The ribbon assembly of claim 12, wherein said oligomer further comprises a polyether polyol residue.

16. The composition of claim 10, further comprising gamma-mercaptopropyl trimethoxysilane.

17. The coated optical fiber of claim 11, wherein said composition further comprises gamma-mercaptopropyl trimethoxysilane.



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18. The ribbon assembly of claim 12, wherein said composition further comprises gamma-mercaptopropyl trimethoxysilane.

19. The coated optical fiber of claim 11, wherein the ratio of the change in length of said inner primary coating to the change in length of said outer primary coating is less than 2 when said coatings are heated from 25° C. to stripping temperature.

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20. The ribbon assembly of claim 12, wherein the ratio of the change in length of said inner primary coating to the change in length of said outer primary coating is less than 2 when said coatings are heated from 25° C. to stripping temperature.

\* \* \* \* \*

## CERTIFICATE OF SERVICE

Pursuant to Federal Rules of Appellate Procedure (“FRAP”) 25(a)(2)(D) and 25(c) and the May 17, 2012 Administrative Order Regarding Electronic Case Filing (“Order”), I hereby certify that on this 21st day of October, 2014, I caused true and correct copies of the foregoing **BRIEF OF APPELLANT CORNING INCORPORATED** to be electronically filed with the Clerk of the Court using CM/ECF, which will send notification of such filing to all registered participants.

Pursuant to ECF-10 of the Order, six (6) paper copies of the foregoing **BRIEF OF APPELLANT CORNING INCORPORATED** will be filed with the clerk within five (5) days of the Court’s acceptance of the **BRIEF OF APPELLANT CORNING INCORPORATED** in ECF.

Pursuant to FRAP 25(c) and ECF-6, true and correct copies of the foregoing **BRIEF OF APPELLANT CORNING INCORPORATED** were electronically served on counsel listed below on this 21st day of October, 2014.

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## CERTIFICATE OF COMPLIANCE

Pursuant to Federal Rule of Appellate Procedure 32(a)(7)(C), I hereby certify on this the 21st day of October, 2014, that the foregoing **BRIEF OF APPELLANT CORNING INCORPORATED** complies with the relevant type-volume limitation of Federal Rule of Appellate Procedure 32(a)(7)(B). This brief is typed in Times New Roman (14 point) and contains 11,561 words according to the Microsoft Word 2010 system used to prepare it excluding those items exempted by Federal Rule of Appellate Procedure 32(a)(7)(B)(iii) and Federal Circuit Rule 32(b).

Dated: October 21, 2014

By: /s/Michael L. Goldman  
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